NMBAQC's Best Practice Guidance

Particle Size Analysis (PSA) for Supporting Biological Analysis

Author: Claire Mason

NE Atlantic Marine Biological AQC Coordinating Committee

NMBAQC's Best Practice Guidance
Particle Size Analysis (PSA) for Supporting Biological Analysis

Table of Contents

List of Figures

List of Tables

Version	Date	Details of changes made
1	2011	Original version
2	21/01/2015	5.1 Sample preservation – 'must freeze' changed to 'should freeze'.
3	18/01/2016	5.4 All sediment received must be analysed. 5.4.2 Laser sizing 3 X 3 replicates – 'must' changed to 'should' complete 3 X 3 replicates with explanatory text; addition of subsampling guidance; use of 2mm mesh for screening if laser instrumentation allows is acceptable. 5.4.6-Addition to indicate all sample material to be kept for quality assurance purposes (at least one year).
4	03/03/2022	Appendix 11.4 NMBAQC PSA Laboratory workshop notes (7th December 2017) and Appendix 11.5 NMBAQC PSA Data Standards workshop notes (22nd June 2018) added. Where appropriate the main sections have been updated to reflect information added from these workshops (references included in these sections),

1 Summary

Standard procedures are described for sampling and sediment particle size analysis (PSA). They are divided into sample collection, sample analysis, data recording and quality assurance. Recommendations are made at the end of each chapter, and these are combined in the concluding chapter. Competent monitoring authorities (CMAs) completing PSA in support of biological analysis for CSEMP and WFD monitoring programmes must adopt these recommendations, as indicated in the Green Book (CSEMP Sampling Procedural Guidelines: Appendix 9).

https://www.cefas.co.uk/publications/greenbook/greenbookappendicesv15.pdf

2 Introduction

Over the 15 years of the NMBAQC's Particle Size component, some anomalies in participants' results have raised questions about the methods that are used by different laboratories to conduct Particle Size Analysis (PSA). A questionnaire sent out to participants in June 2008 confirmed these suspected differences with substantial variation in the methods of sediment sample collection, analysis and reporting between the laboratories who are involved in national level marine monitoring in the UK (e.g. CSEMP and WFD programmes).

Following the review of the questionnaire results, a workshop was held at Cefas, Lowestoft in February 2009 which brought together biologists and sedimentology analysts from the UK's Competent Monitoring Authorities (CMAs) and commercial laboratories. The aim of this workshop was to enable organisations to discuss the different methodologies used, and explore the options/implications of the NMBAQC recommending some 'best practice' methods which should be followed by all laboratories involved in PSA for supporting biological analysis in the CSEMP and WFD marine monitoring programmes. Proceedings from the workshop are available (Addison, 2009).

Since February 2009, workshop participants have worked together and developed a standardised PSA method. This report gives best practice guidance for completion of PSA in support of biological analysis. The guidance is split into the following four sections: Sample Collection (chapter 4); Sample Analysis (chapter 5),

Data Reporting (chapter 6) and Quality Assurance (chapter 7) with an Appendix containing supporting evidence.

Subsequently further workshops have been held:

- PSA workshop 2014 focussing on subsampling methodology (now added to this guidance).
- PSA laboratory workshop 2017 focussing on laser sizer methodology, as well as highlighting presence of low levels of asbestos in sediment samples and implications for laboratory analyses (now added to this guidance).
- PS Sediment Particle Size Data Standards meeting 2018 focussing on sediment data quality and metadata requirements (now added to this guidance).

The terminology used in this report is split into two levels:

1. If a recommendation includes the term '**must**' then this is mandatory for organisations completing PSA that is contributed to UK monitoring programmes.
2. If a recommendation includes the term '**should**' then this is mandatory where practicable for these organisations.

3 Abbreviations

CMA	Competent Monitoring Authority
CSEMP	Clean Seas Environmental Monitoring Programme
CV	Coefficient of variation
JCOP	Joint code of Practice
MERMAN	The Marine Environment Monitoring and Assessment National database
MSFD	Marine Strategy Framework Directive
NMBAQC	NE Atlantic Marine Biological Analytical Quality Control
NMMP	National Marine Monitoring Programme (now CSEMP)
PACQS	Particle Characterisation Quality Assurance Proficiency Scheme (now no longer running)
PSA	Particle size analysis
PSD	Particle size distribution
QA	Quality Assurance
QC	Quality Control
SOP	Standard Operating Procedure
WFD	Water Framework Directive

4 Sample Collection

Sample collection guidance is given for sites where samples are soft sediments (muds, muddy sands, sandy muds, sands) having a predominant particle size diameter of less than 10mm. This criteria is acceptable for current CSEMP sites, but **must** be reviewed if monitoring programmes are redesigned to include coarser substrates. For coarser sediments different sampling gear, different subsampling of sediment for PSA, and larger volume of sample will be required.

4.1 Source of sediment sample

The best practice protocol for macrobenthic grab sampling for CSEMP and WFD is to collect macrobenthic samples from a standard 0.1m^2 Day grab (following Proudfoot *et al*., 1997). This ensures all macrobenthic samples collected around the UK are of a comparable area/volume of seabed. In order to ensure the integrity of macrobenthic samples (for macrobenthic infaunal analysis) all supporting parameters (sediment and chemistry) **must** be collected from a separate grab. Collection of a sediment sample from a separate grab to the biological sample is specific to CSEMP and WFD monitoring programmes, and continuation of previous sediment collection methods such as from the same grab as the biology is acceptable depending on the purpose of the work being completed.

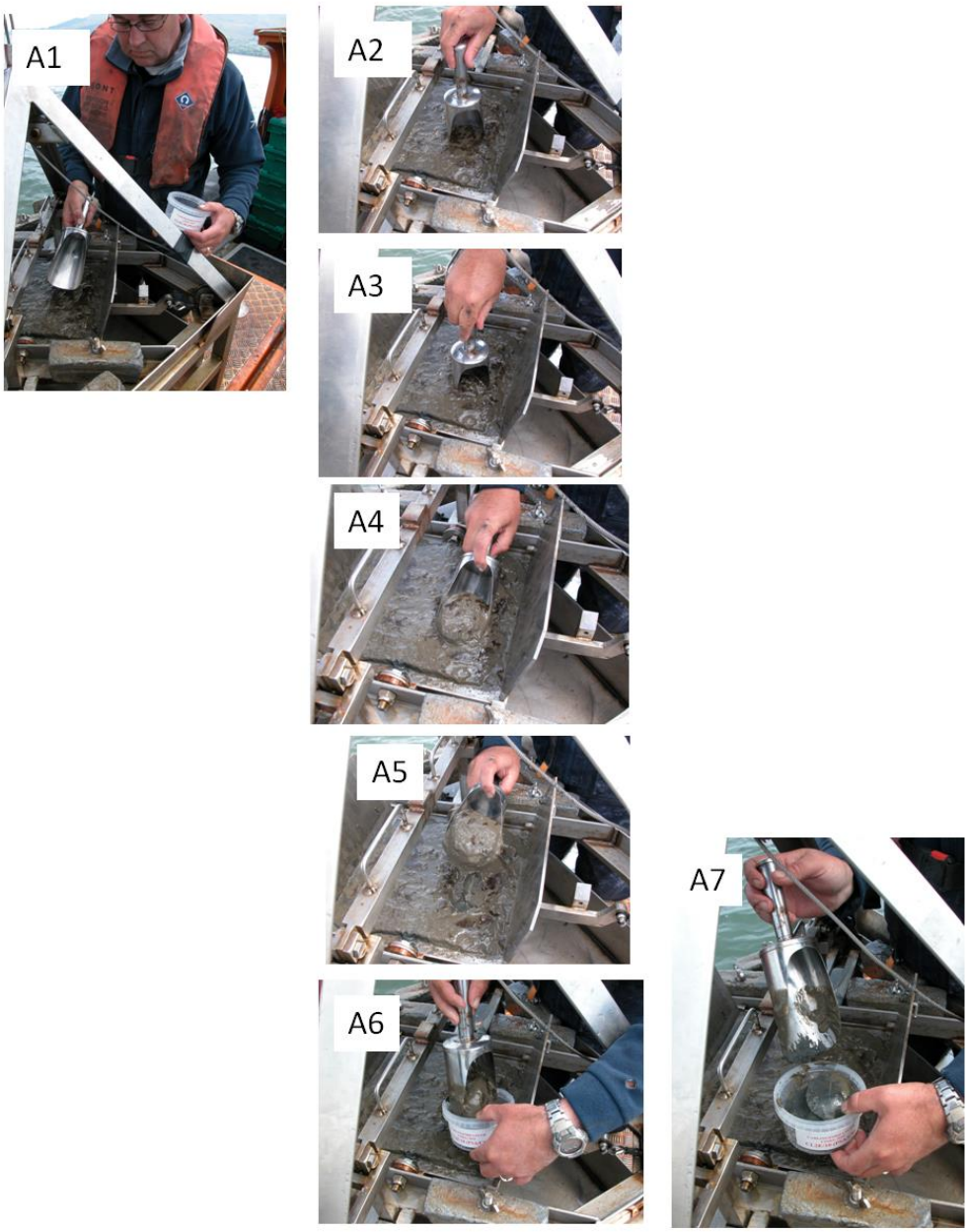
Given that sediment samples are collected from separate grabs to the biology grabs, it is important that each grab is subject to a visual assessment to ensure that the sediment type in the grab is representative of the sample site and biology grabs which have been collected. A visual sediment description (recorded on a sample log sheet) along with a photograph of the sediment surface within the grab **must** be collected for each sample. Depth of sample (from the centre of the Day grab) or volume (calculated from depth of sample multiplied by dimensions of grab) **must** be recorded, with a minimum acceptance depth of 5cm (or equivalent volume of 5cm depth). Grab samples **must** be rejected if they suffer from insufficient depth (less than 5cm), washout, or unequal bite.

4.2 Method of sample collection

Sediment samples for PSA **must** be collected as a depth integrated ‘core’ from a Day grab in order to characterise the sediment which benthic infauna inhabit. A 250ml scoop **must** be inserted vertically into sediment as far as the grab base and rotated to create a core-like plug.

Figure 4.1 shows the removal of a depth integrated ‘core’ using a 250ml scoop, in a series of photos from A1 to A7.

Figure 4.1 Removal of a depth integrated 'core' from Day Grab sediment sample using a 250ml scoop



4.3 Sample volume collected

Sample volume required to ensure a representative PSA is dependant on the particle sizes present at the site concerned. In a muddy sediment, a relatively small volume (100 ml) is required for analysis because within this amount there will be millions of individual particles. In coarse, gravelly samples, a much greater volume of sediment is required to achieve a similar number of particles (British Standards Institution, 1996; Passchier, S., 2007). For

practical purposes, this may not be possible and in this case a 500ml subsample can be used (Boyd, S, 2002). Therefore a minimum volume of sediment of 100ml **must** be collected for PSA (refer to note in chapter 4).

4.4 Removal of conspicuous fauna from sediment samples in the field

Field staff **should** inspect the sediment surface and remove any large/conspicuous (>2 cm) live marine fauna. This includes any live vertebrates (e.g. small fish) or invertebrates (e.g. crustaceans, polychaetes, echinoderms, molluscs etc.). Systematic removal of live marine fauna will be done during laboratory analysis (chapter 5.2).

The presence of large/conspicuous fauna and plant material from a grab which the sediment sample was taken **should** be recorded for each sample.

Shell debris (e.g. empty mollusc shells or pieces of urchin test's, or worm tubes) **must not** be removed from the sediment sample, as these are considered a part of the marine sediment structure.

4.5 Summary recommendations for Sample Collection

Table 4.1 contains all the recommendations given in relation to sample collection. Details of evidence, in terms of experiments (presented in Appendix 11) as well as references are included alongside each recommendation where appropriate.

Table 4.1 Sample Collection Recommendations

Chapter reference	Sample collection	Evidence: Reference/ Appendix
4	Sampling collection guidance **must** be reviewed if monitoring programmes are redesigned to include coarser substrates.	-
4.1	Macrobenthic samples **must** be collected from a standard 0.1m^2 Day grab	Proudfoot *et al*., 1997
4.1	All supporting parameters (sediment and chemistry) **must** be collected from a separate grab.	Appendix 11.1
4.1	A visual sediment description along with a photograph of the sediment surface within the grab **must** be collected for each sample.	Appendix 11.3.3
4.1	Grab samples **must** be rejected if they suffer from insufficient depth penetrated (<5cm), washout or unequal bite.	Cooper, K and Rees, H, 2002
4.2	Sediment samples for PSA **must** be collected as fully depth integrated cores.	Appendix 11.1
4.2	The depth (or volume) of sediment in the grab (from the centre) **must** be recorded, with a minimum acceptance depth of 5cm (or equivalent volume of 5cm depth).	-
4.2	A 250ml scoop **must** be inserted vertically into sediment as far as the grab base and rotated to create a core-like plug.	-
4.3	A minimum volume of sediment of 100ml **must** be collected at each sample site for PSA.	Boyd, S., 2002; British Standards Institution, 1996; Passchier, S.,

		2007
4.4	Field staff **should** inspect the sediment surface and remove any large/conspicuous (>2 cm) live marine fauna	-
4.4	The presence of large/conspicuous fauna and plant material from a grab which the sediment sample was taken **should** be recorded for each sample.	-
4.4	Shell debris **must not** be removed from the sediment sample.	-

5 Sample Analysis

5.1 Sample storage and preservation prior to laboratory analysis

Samples **should** be kept in a sealed plastic container or bag, and frozen as soon as possible.

Sample containers **should** be arranged so that containers are stored upright to avoid leakages. If samples can not be directly placed into a freezer, then a cool box can be used for duration of sampling episode if no refrigeration facilities are available.

The time frame between samples being collected and frozen **should** be minimised, with a maximum time before freezing of 24 hours, and a maximum freezer storage time of 5 years.

5.2 Removal of conspicuous fauna and flora from sediment samples in the laboratory

When conducting PSA of sediment samples, laboratory staff **should** remove any conspicuous marine fauna (>1mm) which appear to have been alive at the time of sampling. This includes any vertebrates (e.g. small fish) or invertebrates (e.g. crustaceans, polychaetes, echinoderms, molluscs etc.). Any shell debris (e.g. empty mollusc shells or pieces of urchin test's, or worm tubes) **must not** be removed from the sediment sample, as these are considered a part of the marine sediment structure.

Likewise any flora, such as red coralline algae, hydroids, and sabellaria, **must not** be removed if they constitute an integral component of the sediment. Presence of flora **should** be recorded in the sediment description.

5.3 Sample preparation

PSA methods can use various possible pre-treatments prior to analysis. These include oven or freeze drying the sediment, removing organics from the sediment, use of dispersant to dis-aggregate sample, removing shell from the sediment by acid digest, as well as various combinations of these.

Various pre-treatments were tested by NIEA (Appendix 11.1). This work has shown that oven drying sediment causes the aggregation of particles in muddy sediments (>5%mud). For these reasons such sediments **should not** be oven dried prior to particle size analysis.

Pre-treatment of samples with hydrogen peroxide to remove organics caused differences in the PSDs measured, compared with samples not pre-treated in the NIEA experiment.

However, this will be different for different sediments and therefore for some sediment (with no organic content) there will be no difference in the PSD measured, as shown in Cefas experiment (Appendix 11.1.2). Therefore, if organics are not removed, variability in the PSD must be expected in relation to the organic content. The organic content is considered to be an integral component of the sediment and **must not** be removed prior to PSA.

The sample needs to be fully dispersed, prior and during laser analysis. There are minimal adverse effects if a chemical dispersant is used, and their use can help with clay/sticky samples. While treatment of samples with dispersant did not cause differences in PSDs measured compared with samples not pre-treated in the NIEA experiment, based on the evidence provided in 2017 workshop (Appendix 11.4), use of a chemical dispersant is acceptable. Addition of 1ml of 3% sodium hexametaphosphate was tested for 3 samples and results are presented. Details of dispersant concentration and amounts, if used, **must** be included within sample metadata to allow measurement comparability.

Shells in the sediment **must** be included in PSA as these are considered an integral part of the marine sediment structure.

5.4 Recommended PSA methodology

This methodology has been produced to ensure consistency between CMAs participating in CSEMP and WFD monitoring programmes. Standard procedures such as those contained within BS1377 (British Standards Institution, 1996) were considered. BS1377 is based on sieve and pipette/sedimentation methods. Most laboratories now measure particle size by laser diffraction as this is less labour intensive, gives high resolution results, and is more efficient.

The methodology is developed from that required to complete PSA of diamictons (mixed sediment including gravel, sand and mud content) (refer to Appendix 11.2.1). These sediments represent the most difficult to measure due to their broad distribution. Sieve and laser diffraction methods are used.

It should be noted that for some sediment types such as clean gravel (sieving only) and sands/sandy muds/muddy sands (laser diffraction only) it is possible to measure using one technique only and therefore avoid merging issues. Merging issues arise because sieve and laser diffraction methods measure particle size differently. Sieving records a particle using the two shortest dimensions, while laser diffraction measures the particle equivalent to a sphere of the volume measured. Therefore particles measured by laser diffraction are bigger than the same particles measured by sieves. The closer the particle is to a sphere the closer the similarity between the two measurements is. Examples of samples measured by both sieve and laser methods to allow comparison and highlight such merging issues are included in Appendix 11.2.3.

In addition to this, laser diffraction methods may underestimate clay content (Appendix 11.1.2 test c) and therefore may not be appropriate for use if accurate clay concentrations are required, for example to link to contaminant data.

However, taking these limitations into account, this is the defined PSA methodology all CMAs **must** use for CSEMP and WFD monitoring programmes, in support of biological

analysis**. If a CMA wishes to use an alternative method they must submit this methodology to the NMBAQC and request approval before completing PSA on any CSEMP/WFD sediment samples.** The methodology can be applied to all sediment types measured (although the sample collection limitations should be taken into account (chapter 4). All sediment >1mm (including 1mm) is measured using sieving, and all sediment <1mm is measured by laser diffraction. This consistency will allow sediments of all types to be measured, and ensure results produced by different laboratories will be able to be used to assess monitoring trends across a wide spatial scale.

A description of each step in the PSA methodology is given below to be used in conjunction with a flow chart in Figure 5.1 (based on flow chart produced by Pye,K and Blott,S, 2009, in Appendix 11.2.1).

Figure 5.1 Flow chart describing steps involved in recommended PSA methodology

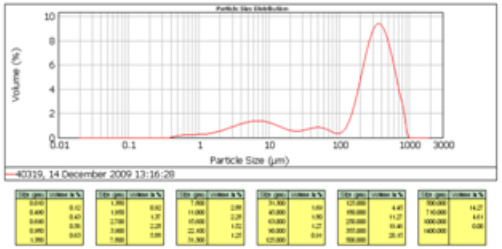
Removal of representative subsample for laser diffraction measurement

5.4.1. Sediment sample- complete a visual assessment

5.4.2. Complete laser diffraction of representative subsample (using 1mm screen)

5.4.3. Wet split remaining sediment sample at 1mm

OVEN-DRY



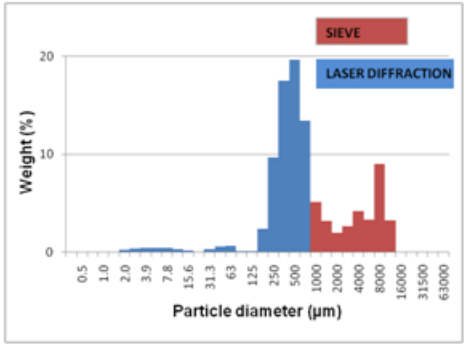
Example of laser diffraction measurement

5.4.4. Record dry weight of <1mm sediment

5.4.5. Dry sieve >1mm sediment at 0.5 ϕ intervals

Oven-dried sediment >1mm

5.4.6. Merge the sieve and laser together to produce a full PSD at 0.5ϕ intervals



Example of full PSD (merged sieve and laser diffraction data)

PSA Standardised Methodology

All the sediment from each sample must be analysed. Generally the sample size is small (<1kg) and therefore it is important, particularly if gravel particles are present, that all the sample is quantified.

5.4.1 Visual assessment

Prior to PSA a sample description **should** be recorded. This should be as standardised as possible, using least dominant to most dominant sediment type present, such as muddy sand, which is sediment consisting predominantly of sand with some mud present. The description **should** include details regarding composition, for example, whether it is shelly. Details of conspicuous fauna (thought to be alive at time of sampling) that removed from the sediment **should** be recorded and noted (chapter 5.2).

5.4.2 Laser diffraction analysis of <1mm sediment fraction

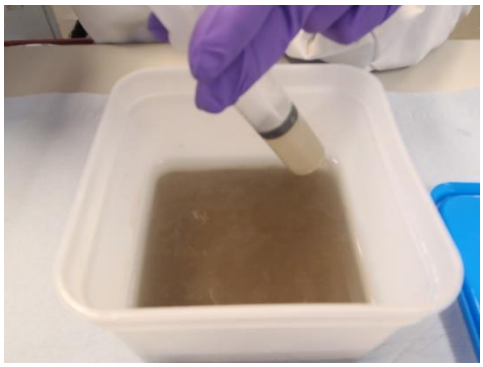
Prepare and analyse a representative subsample of the bulk sample using laser diffraction. Pass the sample through a 1mm mesh prior to analysis. If laser instrumentation allows, screening at 2mm and then splitting the data at 1mm is acceptable. Screening at 2mm is desirable as it means there is better chance of achieving all PS analysis using the laser method, reducing the need for merging data. Also sedimentologically, this means only one method is used for sands. However, as discovered during the workshops, there are some laser sizers that may become damaged if sediments are screened at 2mm. Therefore this is why this methodology has advocated to screen sediments at 1mm.

In 2014, further subsampling guidance has been produced. This covers removal of representative subsample of the bulk sample, followed by removal of laser subsample for laser analysis.

The volume of the laser subsample removed from the bulk sample should be approximately 100ml. This will give enough sample for replicating laser analysis, as well as ensure there is enough sample for quality assurance purposes (Section 5.4.6). This sample should be kept in the fridge during analysis period and can be placed in the freezer for long term storage.

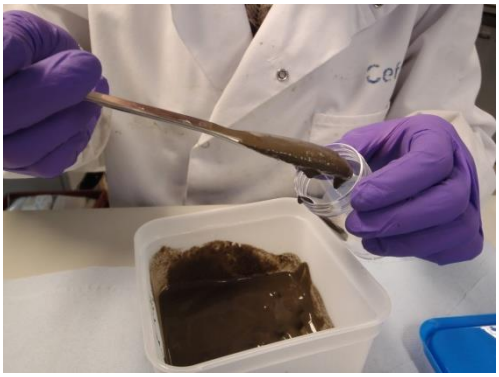
Subsampling from whole sample:

1. Siphon off any clear water before attempting to remove a subsample. The sample will need to have been standing until the fine sediment has completely settled.



2. When as much as possible of the water has been removed, mix the sample thoroughly until it is completely homogenised. Make sure that the sediment is mixed into the corners and bottom of the container.

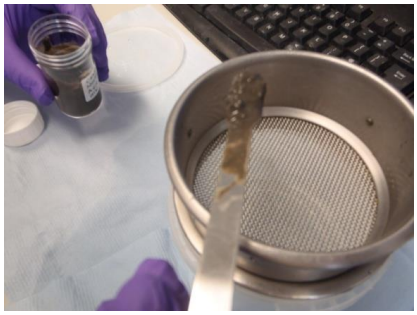
3. Take a representative sub-sample with the spatula and place into a labelled laser pot. Do not add any water to the sample during this process.



Subsampling from laser subsample for laser analysis:

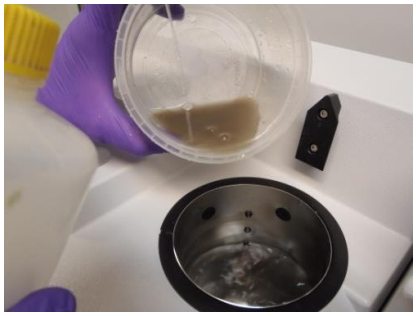
1. Gently homogenise the sample thoroughly in the laser pot with a small spatula.

2. Perform a quick visual assessment of the sample and determine expected result.

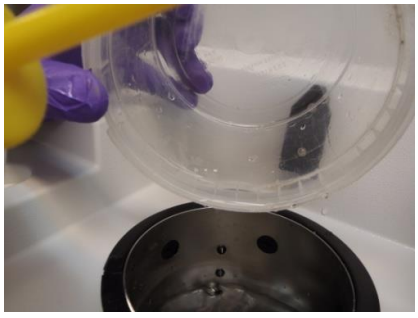


3. Take a small representative subsample from the laser pot and place on a 1mm sieve.

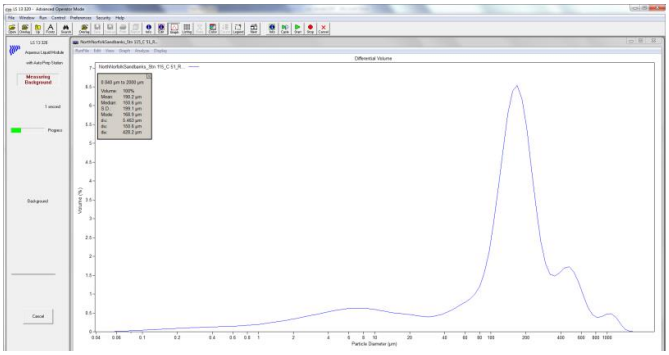
4. Wash the sample through the sieve using a wash bottle, using as small amount of water as possible.



5. Pour all of the <1mm sample into the sample chamber, and rinse the pot out with a wash bottle.



6. Check the results file against expected result (step 2).



Ultrasound (usually completed in the instrument) should be used to assist dispersion of sediments prior to laser diffraction analysis. Use of ultrasonics during laser analysis (as well as before) may help minimise agglomeration of particles (Appendix 11.4).

Laboratories should develop a SOP for sediments based on testing samples, based on experience and instrument manufacturer guidance. Advice can be requested through the NMBAQC.

Complete laser diffraction analysis of three subsamples. Analyse each subsample for three measurements by laser diffraction. Confirm laser methodology is repeatable over a range of sediment types. A lower number of replicates, both in terms of separate sample runs, as well as number of instrument runs, is acceptable providing the laboratory are confident in repeatability of results. However, it is still expected that for at least one sample in 10 a separate subsample is run, and comparison of results for these is checked prior to finalising results. This was confirmed during 2017 Laboratory workshop (Appendix 11.4).

The minimum laser run duration **must** be at least 60 seconds to cover the broad range of particle sizes potentially present in samples being analysed (Appendix 11.4).

For polydisperse, mixed sediments, then maximising obscuration (instrument dependent) helps to ensure sand sized particles are not masked by finer particles, but care should be taken not to introduce multi-scattering effects (Appendix 11.4).

Different laser optical models give different results. The absorption index should be set as duration **must** be at least 60 seconds to cover the broad range of particle sizes potentially present in samples being analysed (Appendix 11.4). In the 2017 NMBAQC PSA workshop (Appendix 11.4) three different optical models:

- Mie theory with a refractive index (R.I.) of 1.55 and an absorption index (A.I) of 0.01
- Mie theory R.I 1.55, A.I 0.1
- Fraunhofer

were tested on two different instruments:

- Malvern Mastersizer 3000
- Beckman Coulter LS13-320.

The Mie theory optical model with A.I of 0.01 produced strange artefacts (both instruments) and therefore **should** not be used. The other two optical models (AI 0.1 and Fraunhofer) both performed better. Further work is needed with a wider range of sediment types to confirm the most comparable model to reduce difference in results between different laser sizer instruments.

If there is no sediment >1mm (left on the 1mm mesh), then no further analysis is required.

5.4.3 Wet splitting sediment sample at 1mm

Wet split the remaining sediment at 1mm. This can be done using a 1mm sieve on a mechanical wet sieve shaker (for example, a Retsch AS 200), or by placing a 1mm sieve/mesh over a bucket. The sediment is placed on the 1mm sieve/mesh and then water is used to flush sediment < 1mm through the sieve/mesh.

Care must be taken not to overload the sieve/mesh or it will become blocked and sediment <1mm will not be able to get through it.

Water should run clear to show no fine sediment is still present on the top of the sieve/mesh.

Wash sediment from the top of the 1mm sieve/mesh into a container. Oven dry the >1mm sediment if this sediment is to be dry sieved, and once dried leave to cool.

Alternatively the sediment can be wet sieved with sieve sizes defined in Table 5.1. The dry weight of sediment in each sieve is then recorded as for dry sieving (Section 5.4.5).

5.4.4 Weight of <1mm sediment fraction

Leave sediment <1mm to settle out from the water over a 24 hour period. Siphon off the clear water from above the sediment surface and then wash the <1mm sediment into a pre-weighed container. Dry the <1mm sediment and record weight. Place the dried sediment in a labelled bag and keep for quality assurance purposes (Section 5.4.6).

5.4.5 Dry sieving >1mm sediment fraction

Dry sieve the sediment >1mm at 0.5φ intervals. Record weight retained by each sieve. Sieve sizes (corresponding to φ scale) that must be used are listed in Table 5.1. If the sediment contains a large proportion of sediment of one sieve size this may cause 'over-loading'. In this case, it is necessary to split the sample and analyse each part separately, combining the data at the end (Table 7.2). Place the dried sediment in a labelled bag and keep for quality assurance purposes (Section 5.4.6).

5.4.6 Quality assurance of PS results

Laboratories **must** keep components of samples (laser sub-sample (5.4.2), dry sieve >1mm fraction (5.4.5), and weigh-back <1mm fraction (5.4.4)) so that reanalysis is possible for quality assurance purposes, within 1 year of analysis. The NMBAQC run a PS-own sample module. Participants are asked to supply a dataset, from which 3 samples are selected. These 3 samples are re-analysed and these results are compared with the original dataset.

Table 5.1 Sieve sizes at 0.5φ intervals

φ value	Equivalent sieve size (mm)
-6	63
-5.5	45
-5	31.5
-4.5	22.4
-4	16
-3.5	11.2
-3	8
-2.5	5.6
-2	4
-1.5	2.8
-1	2
-0.5	1.4
0	1

5.4.7 Merging of sieve and laser diffraction data

After completing QA of sieve and laser data (Section 7), merge the sieve and laser data together to produce a complete PSD at 0.5φ intervals, by completing the following calculations. A worked example of these calculation steps is also included in Appendix 11.2.4.

Remove any laser data >1mm, and then rescale it to 100%.

Convert laser data into weights (using total weight of <1mm sediment – (chapter 5.4.4**)** + dry sieve pan (sediment <1mm) (Section 5.4.5)).

Use sieve weights for sediment >1mm including 1mm fraction, and derived laser weights for sediment <1mm.

Produce a merged PSD percentage distribution at 0.5φ intervals.

Some laser sizing instruments have modelling software that enables users to add sieve data to the laser data and merge together. For NMBAQC purposes, such modelling software **must not** be used as it may merge the data in a different way and introduce inconsistencies to the data. Laser data **must** be merged with sieve data independently.

5.5 Summary recommendations for Sample Analysis

Table 5.2 contains a summary of all sample analysis recommendations. Details of evidence, in terms of experiments (presented in Appendix 11) as well as references are included alongside each recommendation where appropriate.

Table 5.2 Sample Analysis Recommendations

Chapter reference	Sample Analysis Recommendations	Evidence: Reference/ Appendix
5.1	Samples **should** be kept in a sealed plastic container or bag, and frozen as soon as possible.	-
5.1	Sample containers **should** be arranged so that containers are stored upright to avoid leakages	-
5.1	The time frame between samples being collected and frozen **should** be minimised, with a maximum time before freezing of 24 hours, and a maximum freezer storage time of 5 years.	-
5.2	Laboratory staff **should** remove any conspicuous marine fauna (>1mm) which appear to have been alive at the time of sampling.	-
5.2	Any shell debris **must not** be removed from the sediment sample	-
5.2	Plant material **must not** be removed from the sediment sample .	-
5.3	Muddy (>5% mud) sediments **should not** be oven dried prior to particle size analysis.	Appendix 11.1.1 test c
5.3	Organic matter **must not** be removed prior to PSA.	Appendix 11.1.1 test c ; Appendix 11.1.2 test c, Appendix 11.1.3
5.3	*Previously, NMBAQC PSA guidance stated that dispersants **should not** be used for PSA.* Subsequent evidence provided supports use of dispersants. Details of dispersant concentration and amounts, if used, **must** be included within sample metadata to allow measurement comparability.	Appendix 11.1.1 test c, Appendix 11.4
5.3	Shells in the sediment **must not** be removed from sediment prior to PSA.	-
5.4	All CMAs **must** use the PSA standardised methodology defined.	-
5.4	**Any CMA using an alternative PSA method must submit methodology and have this approved by the NMBAQC before completing any PSA on CSEMP/WFD sediments**	-
5.4	All the sediment sample **must** be analysed.	
5.4.1	Visual Assessment: A sample description **should** be recorded.	-
5.4.1	Details of conspicuous fauna (thought to be alive at time of sampling) that removed from the sediment **should** be recorded and noted	-
5.4.2	Laser diffraction: The minimum volume of sediment for laser analysis **should** be 100ml.	
5.4.2	Laser diffraction: If laser instrumentation allows screening at 2mm and then splitting the data at 1mm is acceptable.	Appendix 11.4
5.4.2	Laser diffraction: At least 1 in 10 laser subsamples **must** be analysed twice, and where samples are unstable more replicates may be required. Original guidance stated that *3 subsamples must be analysed, each for 3 measurements, giving a total of nine measurements for each sample measured.* This is good practice when setting up new methodology as was	ISO 13320, 2009

	the focus at the time the original guidance was produced. However, once a laboratory is confident that their methodology is stable then this is unnecessary.	
5.4.2	Laser diffraction: The minimum laser run duration must be at least 60 seconds.	Appendix 11.4
5.4.2	Laser diffraction: Recommended to use maximum obscuration (instrument manufacturer specific) possible for polydisperse sediment samples.	Appendix 11.4
5.4.2	Laser diffraction: Optical model. A.I of 0.01 **should not** be used.	Appendix 11.4
5.4.6	Laboratories **must** keep sample material, for quality assurance purposes, for at least 1 year.	-
5.4.7	Laser sizer modelling software **must not** be used to merge sieve and laser data. Laser data **must** be merged with sieve data independently.	Appendix 11.2.4

6 Data Reporting

Previously there was confusion regarding the statistics required for data submission to MERMAN. Several different methods exist that can be utilised to generate sediment statistics (Appendix C). In addition to this, most of the statistical parameters generated assume the sediment is normally distributed and is unimodal. In reality, many sediments are bi-modal, as well as polymodal. Statistics calculated for such distributions are therefore meaningless and **should not** be calculated or used for interpretation of sediment data.

Therefore, all CMAs must submit PSD data to MERMAN at 0.5φ intervals as defined by PSA standardised methodology. This will enable data requestors to generate derived parameters for the purpose required, and ensure consistency in calculation used. Gradistat (an Excel based software package, produced by Blott, S, 2001) is freely available (download from http://www.kpal.co.uk/gradistat.html. Gradistat can be used to calculate most standard sedimentological statistical parameters, taking into account the limitations of these when considering bimodal/polymodal PSDs. It can also be used as a cross-reference to in-house automated calculations.

Sediment descriptions and associated PS methodology details **should** be stored in MERMAN. This should also include sample depth (chapter 4.1).

6.1 Summary recommendations for Data Recording

Table 6.1contains all the recommendations given in relation to data recording.

Table 6.1 Summary of Data Recording recommendations

Chapter reference	Data recording recommendations	Evidence: Reference/ Appendix
6	Full PSD data at 0.5φ intervals **must** be submitted to MERMAN.	-
6	Derived statistical parameters **should not** be calculated for polymodal distributions.	-
6	Derived statistical parameters **must not** be stored in MERMAN.	-
6	Sediment descriptions and associated sample metadata **should** be stored in MERMAN.	-

7 Quality Assurance

All government organisations completing PSA for support of biological analysis must have a Quality Assurance (QA) system, with clear evidence of how this is achieved. QA in marine biology is the systematic examination and evaluation of all aspects of a monitoring programme (from survey design, field methods, laboratory methods, data analysis and storage) to ensure that standards of data quality and comparability between organisations are being met. This in turn provides confidence in the evidence base for policy and decision making (Addison, P, 2010).

UK government organisations have to comply with the Joint Code of Practise (Defra, 2003). Some organisations are accredited by the United Kingdom Accreditation Service (UKAS) to ISO/IEC 17025 (2017). UKAS accreditation increases the confidence of customers that work is performed to a high, internationally recognised standard for the competence of laboratories (namely ISO/IEC 17025). This shows that suitable methods are used and that measurements are traceable to international standards. UKAS is recognised by UK government as the national body for providing accreditation for testing laboratories. The laboratory will be stringently assessed by independent auditors to show that the reality of what is actually happening in the laboratory accords with the laboratory's policy and documented procedures. This will provide confidence to the customer that the method is fit for purpose, leading to fewer disputed results and less need for repeated analysis, thus reducing your costs and increasing your operating efficiency. An improved quality of service will give greater customer satisfaction leading to enhanced business opportunities. (Johns, D, 2010, personal communication).

7.1 General QA requirements

All laboratories completing PSA for CMAs **must** participate in the NMBAQC PSA ring test and should participate in the NMBAQC PSA own sample test. QC data must be provided (for example, laser replicate measurements) with NMBAQC PSA ring test results (Appendix 11.4). They **must** have clear SOPs for methods used. Evidence of routine maintenance and calibration of instrumentation **must** be available. All analysts **must** have a training record, showing competence in all procedures outlined in PSA standardised methodology.

7.2 QA requirements linked to PSA standardised methodology

7.2.1 QA: Visual assessment of the sample (5.4.1)

Visual assessments are subjective. They should be standardised much as possible, and include details regarding composition of the sediment, including presence of shells, organic fragments, any biology (individual species or worm-tubes) and indication of anthropogenic presence (eg glass, paint flecks).

7.2.2 QA: Laser Diffraction (5.4.2)

All laboratories **must** be able to demonstrate quality assurance of laser diffraction results for NMBAQC. Examples of QC measures for laser diffraction methods are included in Table 7.1. Laboratory analysts should be fully trained in laser diffraction analysis. Participation in the Particle Characterisation Quality Assurance Proficiency Scheme (PACQS) is advised as a good scheme to develop experience and understanding of laser diffraction and test competency of analysts.

Table 7.1 QC procedures for Laser diffraction

QC Procedure	Frequency	Defining acceptability of results	Remedial action
Use of internal reference standards. Worked example is given in 11.3	At start and end of every sample batch on a daily basis.	Quality control charts. Acceptable limits can be defined based on average +/- 2 stdev on d(0.1), d(0.5) and d(0.9).	If results outside limits, then repeat standard. If still outside limits, check with a certified reference material. If this fails, contact manufacturer.
Use of certified reference standards Examples include glass beads certified references. Possible to use spare proficiency testing samples as certified reference standards. Useful for competence training.	Completion recommended at least once a month	Results within limits defined on the certificate.	If results fail, repeat. If these fail contact instrument manufacturer.
Completion of several measurements for each sample run completed.	Minimum of three measurements recommended for each sample measured.	Coefficient of variation (CV) of d (0.1), d(0.5) and d(0.9) is less than 3% (defined in ISO 133020). Please note that in reality 3% is on the low side, greater variability being expected for natural sediment samples – a maximum of 20% (based on 3 replicates being measured) should be used as a guide.	If 1 out of the 3 results is very different, remove this outlier and recalculate CV. If all 3 results are different, complete a repeat analysis. If this is different again, after removal of clear outliers, calculate the average.
Completing background and alignment of laser checks	Every sample run	As defined by instrument manufacturer	If background or alignment does not fit expected measurements, take advice from instrument manufacturer.
Complete obscuration checks	Every sample run	Obscuration within 15-20% (or as indicated by instrument manufacturer)	Check results outside limits carefully, using repeat data. Remove from dataset for calculation of average.
Complete optical model checks	Every sample run	Check model is appropriate as advised by instrument manufacturer and instrument manuals.	Amend model so that results valid as advised by instrument manufacturer.
Completion of repeat sample measurements. PSA methodology already states that 3 separate subsamples should be measured.	Minimum of three separate subsamples for each sample measured.	CV (as above) or comparison of profiles	If 1 out of the 3 results is very different, remove this outlier and recalculate CV. If all 3 results are different, complete a repeat analysis. If this is different again, after removal of clear outliers, calculate the average.

7.2.3 QA: Wet split the sediment at 1mm (chapter 5.4.3).

Refer to chapter 7.2.5 for QA associated with sieves. There are no measurable QC measures that can be completed for this part of the method. Bench tests and routine observation of analysts completing this procedure should be completed.

7.2.4 QA: Siphon and weigh back <1mm (chapter 5.4.4)

There are no measurable QC measures that can be completed for this part of the method. Bench tests and routine observation of analysts completing this procedure should be completed.

7.2.5 QA: Dry sieving (chapter 5.4.5)

All laboratories **must** be able to demonstrate quality assurance of dry sieving results for NMBAQC. Examples of QC measures that could be used are defined in Table 7.2.

Table 7.2 QC procedures for Dry sieving

QC Procedure	Frequency	Defining acceptability of results	Remedial action
Weighing sample prior to sieving, and after sieving. Comparing totals (pre-sieving, total sieving, and post-sieving) and resieving if discrepancies noted. Worked example included in Appendix D.	Complete for every sample measured.	Losses of 5% unacceptable (Rhodes, 2001)	Repeat analysis of this sample.
Use of certified reference standards	Every 6 months	Results within limits defined on the certificate.	Repeat analysis and replace sieve if necessary.
Use of internal reference standards. Recommended by Buxton, R (2000).	Every analyst completes analysis of an internal reference standard as proof of competence. Recommend every analyst completes analysis of internal reference sediment every 6 months.	Measurement for each sieve is within defined limits.	Repeat analysis and replace sieve if necessary.
Check weight of sample being measured will not load sieve mesh. If the sieve is overloaded particles will be pushed into the holes of the sieve and stop sieving being effective.	Every sample being sieved.	Maximum per sieve defined in British Standards (1996).	If the sieve has been overloaded, clean and complete visual check. Split sample and reanalyse.
Visual checks for holes and mis-shaped areas in the mesh. Keep a record of these checks for lifetime of sieve.	Every sieve at the start of every batch of analysis on a daily basis.	Visual check	Replace sieves as necessary

7.2.6 QA: Merging of sieve and laser diffraction data (chapter 5.4.7)

Merging calculations **should** be cross checked and verified by a Laboratory Manager. The final merged PSD results **should** be compared with sample photographs and sediment description recorded during sample collection (chapter 4.1), as well as the visual assessment made at the start of the PSA standardised method (chapter 5.4.1).

7.3 Summary recommendations for Quality Assurance

Table 7.3 contains all the recommendations given in relation to quality assurance. Details of evidence, in terms of experiments (presented in Appendix 11) as well as references are included alongside each recommendation where appropriate.

Table 7.3 Summary of Quality Assurance recommendations

Chapter reference	Quality Assurance Recommendations	Evidence: Reference/ Appendix
7	All government organisations completing PSA for support of biological analysis **must** have a QA system, with clear evidence of how this is achieved.	Addison, P, 2010
7.1	All laboratories completing PSA for CMAs **must** participate in the NMBAQC PSA ring test.	Green Book Addison, P, 2010
7.1	QC data must be provided (for example, laser replicate measurements) with NMBAQC PSA ring test results.	Appendix 11.4
7.1	All laboratories completing PSA for CMAs **must** have clear SOPs for methods used.	Addison, P, 2010
7.1	All laboratories completing PSA for CMAs **must** have evidence available of routine maintenance and calibration of instrumentation.	Addison, P, 2010
7.1	All analysts **must** have a training record for procedures defined in standardised PSA method.	Addison, P, 2010
7.2.1	Visual Assessment: The sample description **should** include details regarding composition, for example, whether it is shelly.	-
7.2.1	Visual Assessment: Details of conspicuous fauna (thought to be alive at time of sampling) that removed from the sediment **should** be recorded and noted	-
7.2.2	Laser Diffraction: All laboratories **must** be able to demonstrate quality assurance for NMBAQC	Table 7.1; Appendix 1.1
7.2.5	Dry sieving: All laboratories **must** be able to demonstrate quality assurance for NMBAQC	Table 7.2; Appendix 11.3.1
7.2.6	Merging sieve and laser diffraction data: Merging calculations **should** be cross checked and verified by a Laboratory Manager.	Appendix 11.2.4
7.2.6	Merging sieve and laser diffraction data: The final merged PSD results **should** be compared with sample photographs and sediment description recorded during sample collection (chapter 4.1), as well as the visual assessment made at the start of the PSA standardised method (chapter 5.4.1).	Appendix 11.3.3

8 Conclusions

Recommendations have been made based on experimental evidence (given in Appendices), expert advice and review of references. Table 8.1 contains all recommendations given for sample collection, sample analysis, data recording and quality assurance. These **must** be adopted by all CMAs contributing PSD data in support of biological analysis for CSEMP and WFD monitoring programmes. They will be included in the next update of the Green Book.

It is recognised these will need regular review and updating as new technology and methods superseded the current recommendations. There is a constant need for CMAs and external consultancies completing PSA to maintain links through the NMBAQC.

Table 8.1Combined recommendations given for sample collection, sample analysis, data recording and quality assurance

Chapter reference	Sample collection recommendation
4	Sampling collection guidance **must** be reviewed if monitoring programmes are redesigned to include coarser substrates.
4.1	Macrobenthic samples **must** be collected from a standard 0.1m^2 Day grab
4.1	All supporting parameters (sediment and chemistry) **must** be collected from a separate grab.
4.1	A visual sediment description along with a photograph of the sediment surface within the grab **must** be collected for each sample.
4.1	Grab samples **must** be rejected if they suffer from insufficient depth penetrated (<5cm), washout or unequal bite.
4.2	Sediment samples for PSA **must** be collected as fully depth integrated cores.
4.2	The depth of sediment in the grab (from the centre) **must** be recorded for each sample collected.
4.2	A 250ml scoop **must** be inserted vertically into sediment as far as the grab base and rotated to create a core-like plug.
4.3	A minimum volume of sediment of 100ml **must** be collected at each sample site for PSA.
4.4	Field staff **should** inspect the sediment surface and remove any large/conspicuous (>2 cm) live marine fauna
4.4	The presence of large/conspicuous fauna and plant material from a grab which the sediment sample was taken **should** be recorded for each sample.
4.4	Shell debris **must not** be removed from the sediment sample.

Table 8.1 (continued) Combined recommendations given for sample collection, sample analysis, data recording and quality assurance

Chapter reference	Sample Analysis Recommendations
5.1	Samples **should** be kept in a sealed plastic container or bag, and frozen as soon as possible.
5.1	Sample containers **should** be arranged so that containers are stored upright to avoid leakages
5.1	The time frame between samples being collected and frozen **should** be minimised, with a maximum time before freezing of 24 hours, and a maximum freezer storage time of 5 years.
5.2	Laboratory staff **should** remove any conspicuous marine fauna (>1mm) which appear to have been alive at the time of sampling.
5.2	Any shell debris **must not** be removed from the sediment sample
5.2	Plant material **must not** be removed from the sediment sample .
5.3	Muddy (>5% mud) sediments **should not** be oven dried prior to particle size analysis.
5.3	Organic matter **must not** be removed prior to PSA.
5.3	*Previously, NMBAQC PSA guidance stated that dispersants **should not** be used for PSA.* Subsequent evidence provided supports use of dispersants. Details of dispersant concentration and amounts, if used, **must** be included within sample metadata to allow measurement comparability.
5.3	Shells in the sediment **must not** be removed from sediment prior to PSA.
5.4	All CMAs **must** use the PSA standardised methodology defined.
5.4	**Any CMA using an alternative PSA method must submit methodology and have this approved by the NMBAQC before completing any PSA on CSEMP/WFD sediments**
5.4	All the sediment sample **must** be analysed.
5.4.1	Visual Assessment: A sample description **should** be recorded.
5.4.1	Details of conspicuous fauna (thought to be alive at time of sampling) that removed from the sediment **should** be recorded and noted
5.4.2	Laser diffraction: The minimum volume of sediment for laser analysis **should** be 100ml.
5.4.2	Laser diffraction: If laser instrumentation allows screening at 2mm and then splitting the data at 1mm is acceptable.
5.4.2	Laser diffraction: At least 1 in 10 laser subsamples **must** be analysed twice, and where samples are unstable more replicates may be required. Original guidance stated that *3 subsamples must be analysed, each for 3 measurements, giving a total of nine measurements for each sample measured.* This is good practice when setting up new methodology as was the focus at the time the original guidance was produced. However, once a laboratory is confident that their methodology is stable then this is unnecessary.
5.4.2	Laser diffraction: The minimum laser run duration must be at least 60 seconds.
5.4.2	Laser diffraction: Recommended to use maximum obscuration (instrument manufacturer specific) possible for polydisperse sediment samples.
5.4.2	Laser diffraction: Optical model. A.I of 0.01 **should not** be used.
5.4.6	Laboratories **must** keep sample material, for quality assurance purposes,

	for at least 1 year.
5.4.7	Laser sizer modelling software **must not** be used to merge sieve and laser data. Laser data **must** be merged with sieve data independently.

Chapter reference	Data Recording Recommendations
6	Full PSD data at 0.5φ intervals must be submitted to MERMAN.
6	Derived statistical parameters should not be calculated for polymodal distributions.
6	Derived statistical parameters must not be stored in MERMAN.
6	Sediment descriptions and associated sample metadata should be stored in MERMAN.

Table 8.1 (continued) Combined recommendations given for sample collection, sample analysis, data recording and quality assurance

Chapter reference	Quality Assurance Recommendations
7	All government organisations completing PSA for support of biological analysis must have a QA system, with clear evidence of how this is achieved.
7.1	All laboratories completing PSA for CMAs must participate in the NMBAQC PSA ring test.
7.1	All laboratories completing PSA for CMAs must have clear SOPs for methods used.
7.1	All laboratories completing PSA for CMAs must have evidence available of routine maintenance and calibration of instrumentation.
7.1	All analysts must have a training record for procedures defined in standardised PSA method.
7.2.1	Visual Assessment: A sample description should be recorded
7.2.1	Visual Assessment: The description should include details regarding composition, for example, whether it is shelly.
7.2.1	Visual Assessment: Details of conspicuous fauna (thought to be alive at time of sampling) that removed from the sediment should be recorded and noted.
7.2.2	Laser Diffraction: All laboratories should use all of the defined QC measures are in Table 7.1.
7.2.5	Dry sieving: All laboratories should use all of the defined QC measures in Table 7.2.
7.2.6	Merging sieve and laser diffraction data: Merging calculations should be cross checked and verified by a Laboratory Manager.
7.2.6	Merging sieve and laser diffraction data: The final merged PSD results should be compared with sample photographs and sediment description recorded during sample collection (chapter 4.1), as well as the visual assessment made at the start of the PSA standardised method (chapter 5.4.1).

9 Acknowledgements

Acknowledgements to the NMBAQC, including Tim Mackie (chair of NMBAQC, NIEA), Myles O'Reilly (SEPA), and Keith Cooper (Cefas), supported by Prue Addison (NMBAQC, EA), for directing the approach taken to ensure methodology is fit for supporting biological data.

Acknowledgements are expressed to Ken Pye and Simon Blott (Ken Pye Associates Ltd), Richard Hartley (Plymouth University), Mike Allen (NIEA) and Anne Virden (Malvern) as well as all colleagues including Manuel Nicolaus (Cefas), Thomas Maes (Cefas) and Tracy Maxwell (Cefas). This is essentially a collaborative project and it is hoped to maintain the links made between organisations, and ensure continued development and sharing of best practice.

10 References

Addison, P, 2009, Proceedings of the NMBAQC's Workshop on 'PSA for Supporting Biological Analysis' http://www.nmbaqcs.org/media/4320/nmbaqc%20workshop%20proceedings_final.pdf

Addison, P, 2010, Quality assurances in marine biological monitoring, http://www.nmbaqcs.org/qa-standards.aspx

Blott, S.J., and Pye, K, 2001, Gradistat: A grain size distribution and statistics package for the analysis of unconsolidated sediments, Earth Surface Processes and Landforms, 26, 1237-1248

Boyd, S. (Compiler), (2002) Guidelines for the conduct of benthic studies at aggregate dredging sites, for Department for Transport, Local Government and the Regions, CEFAS, Lowestoft, UK, 117pp

British Standards Institution, (1996) BS1377 British Standards: Part 2: 1996 Methods of test for soils for civil engineering purposes: Classification tests. British Standards Institution, London, UK 61pp

Clarke and Gorley 2006, PRIMER v6: User Manual/Tutorial. PRIMER-E, Plymouth.

Cooper, K.M., and Rees, H.L., 2002, National Marine Biological Analytical Quality Control Scheme (NMBAQC) Review of Standard Operating Procedures, Science Series Aquatic Environment Protection: Analytical Methods, Cefas, Lowestoft (13):57pp.

Defra(2003) Joint code of practice, http://www.defra.gov.uk/evidence/science/how/documents/QACoP-V8.pdf

Green Book:
http://www.cefas.co.uk/publications/scientific-series/green-book.aspx

ISO 13320, 2009, Particle size analysis – laser diffraction methods, 51pp, (available from http://www.iso.org/iso/en/prods-services/ISOstore/store.html)

ISO 17205, 2005, General requirements for the competence of testing and calibration laboratories, 28pp, (available from http://www.iso.org/iso/en/prods-services/ISOstore/store.html)

Passhier, S, 2007, Particle Size Analysis (granulometry) of sediment samples, chapter 14 in Coggan, R., Populus, J., White, J., Sheehan, K., Fitzpatrick, F. and Piel, S. (eds.), Review of Standards and Protocols for Seabed Habitat Mapping. MESH. 210pp.

Proudfoot, R.K., Elliot, M., Dyer, M.F., Barnett, B.E., Allen, J.H., Proctor, N.L., Cutts, N.D., Nikitik, C., Turner, G., Breen, J., Hemmingway, K.L., Mackie, T., 1997, Proceedings of the Humber Benthic Field Methods Workshop, Hull University. Collection and processing of macrobenthic samples from soft sediments; a best practice review. Environment Agency R&D Technical Report E1-135/TR. 140pp.

Rhodes, D., 2001, Sieves- their use and abuse, presented at Training Meeting on Particle Size Measurements, RSC Particle Characterisation Group, 22/03/01 at the Harwell Conference Centre. Abstract available from the RSC Particle Characterisation Group, e-mail address: particle@rsc.org

11 Appendix

11.1 Experimental evidence in support of recommendations

11.1.1 NIEA

11.1.1.1 Introduction

The Northern Ireland Environment Agency (NIEA) completed the following experiments to produce evidence in support of recommendations for PS methodology in support of biological analysis for the NMBAQC.

Aims tested:

a/ the effect of the source of the sediment sample (separate grab or same grab) on the particle size distribution (PSD) measured.

b/ the effect of two methods of sample collection (2 cm surface scrape compared with 5cm depth integrated core) on the PSD measured.

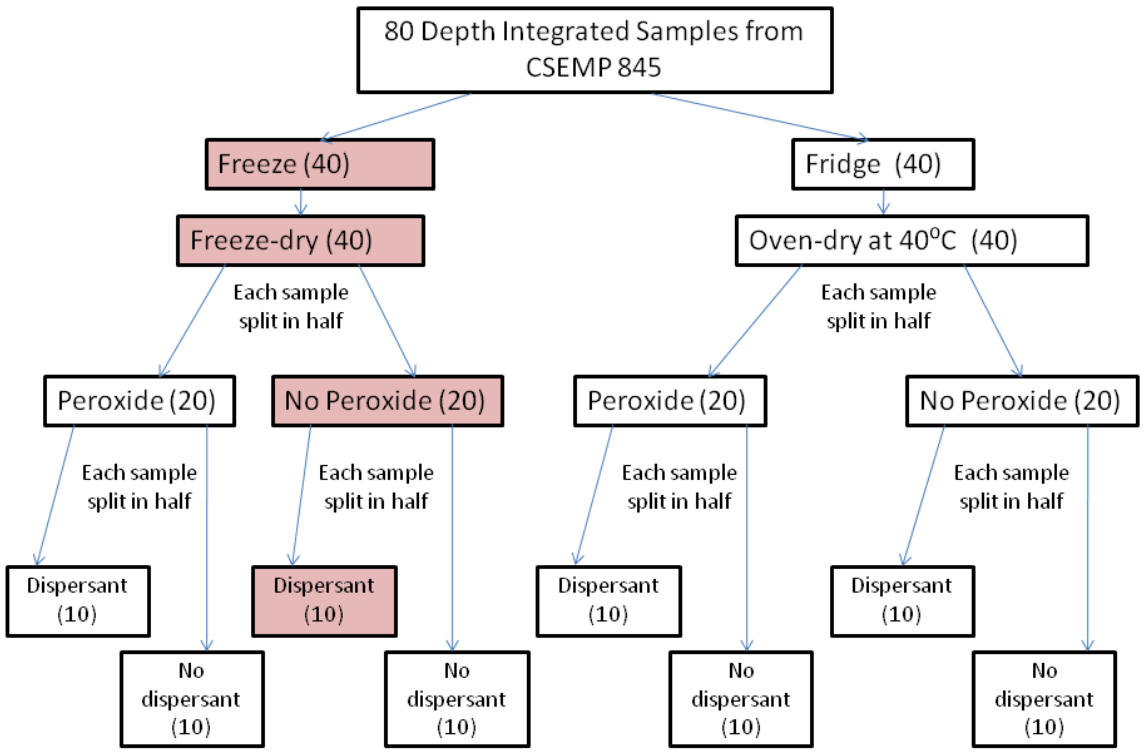
c/ the differences in the PSDs caused by freeze-drying after freezing compared with oven-drying after refrigeration, dispersant compared with no dispersant and organics removal compared with no organics removed.

11.1.1.2 Methods

Eighty samples were collected from 0.1m^2 day grabs to compare the variation between various sample preparation methods, including fridge/oven v freezer/freeze-drying, hydrogen peroxide verses no hydrogen peroxide and dispersant verses no dispersant. The flow chart shown in Figure 11.1 shows the experimental design for these tests. In addition to these 80 samples, two 0.1m^2 day grabs (Grabs 1 and 2) with five depth integrated samples (representing intra-grab variation) were collected to compare with the ten separate 0.1m^2 day grabs (representing inter-grab variation) collected as part of the experimental design shown in Figure 11.1 (coloured pink). A further 10 samples were collected from ten 0.1m^2 day grabs to compare the variation between two sampling methods: surface scrapes and depth integrated cores These samples were all collected from CSEMP 845 on the 12th of June 2009. The volume of sediment collected in each day grab ranged from 6 to 9 litres.

Depth integrated samples were collected with a 250 ml scoop which was inserted vertically into grab sediment as far as the grab base (approx 16 cm) and rotated to create a core-like plug (approx 500 ml wet sample collected). Surface scrape samples were collected with a 250 ml scoop, which were pulled along the sediment surface to a maximum of 2 cm depth (approximately 500 ml wet sample collected).

Figure 11.1 Flow chart showing NIEA experimental design
The methodology indicated by the pink boxes is routinely used for PSA by NIEA, and these 10 results were used to compare with intra-grab measurements and surface scrape measurements. Please note hydrogen peroxide has been shortened to peroxide.



All samples, except the 40 samples collected to test effects of fridge/oven (Figure 11.1) were frozen as soon as they were returned to the laboratory, followed by freeze drying. The 40 samples collected to test effects of fridge/oven were refrigerated and then dried in an oven at 40 degrees Celsius.

In the sample preparation experiment (Figure 11.1), for both sets of samples (fridge/oven-dried and frozen/freeze-dried) half of the samples were treated with 100 ml of 6% hydrogen peroxide to remove organics. The rest of the samples were not treated with hydrogen peroxide.

100 g of sample was dry sieved through 16mm, 8, 4, 2, and 1 mm sieves (20 minutes on a shaker). The <1 mm fraction was retained for laser analysis and was added to the Hydro G section of the Malvern Mastersizer 2000 until obscuration reached 15 % (<0.25 g). 1ml of Calgon (sodium hexametaphosphate 20% solution) dispersant was added to the Hydro G with each sample, except samples being measured without dispersant (Figure 11.1). Measurement cycles commenced following 30 seconds of ultrasound.

Dry sieve and laser data were merged together by normalising laser data to the less than 1mm sieve percentage for each sample.

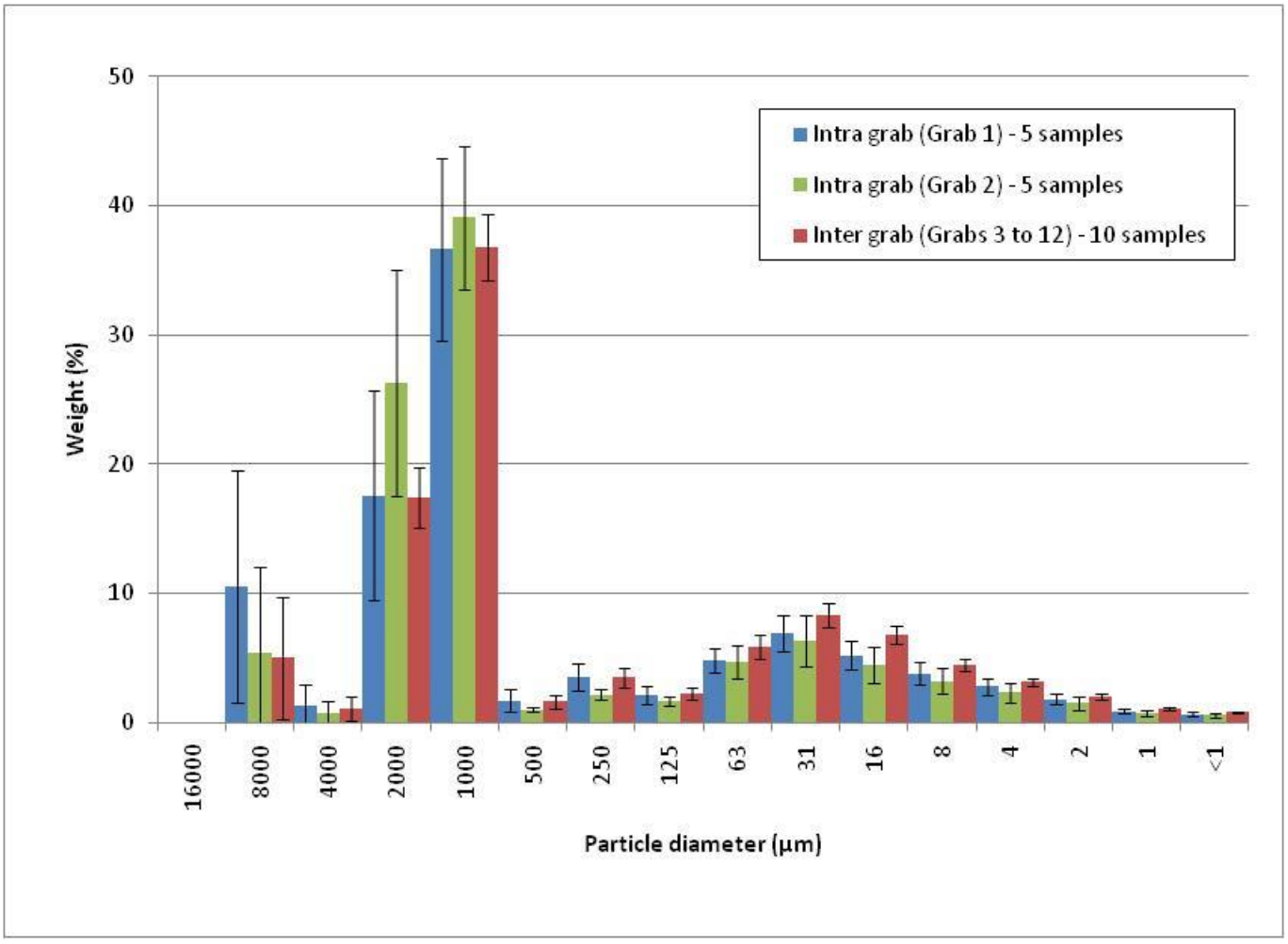
11.1.1.3 Results

The sediments are described as gravelly muddy sands and muddy sandy gravels. They all have a primary mode of 1500µm.

a/ the effect of the source of the sediment sample (separate grab or same grab) on the particle size distribution (PSD) measured

The average intra-grab particle size distribution profiles of Grabs 1 and 2 are compared to the average inter-grab particle size distribution profiles of Grabs 3-12 in Figure 11.2 Figure 11.2. Generally the profiles from each source (intra grab 1, intra grab 2 and inter-grabs 3-12) are well matched.

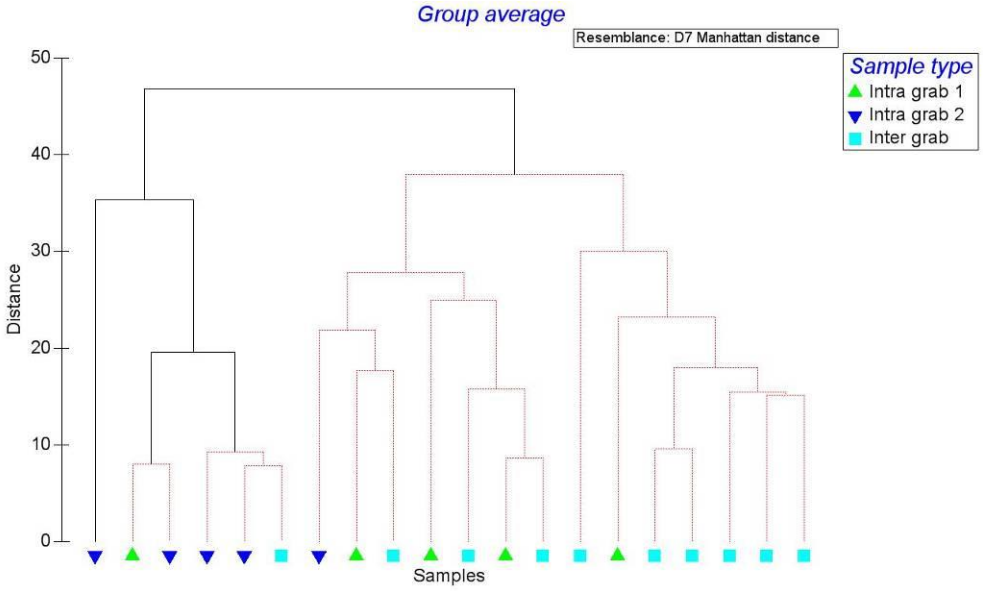
Figure 11.2 Average PSDs of intra and inter grab samples
Intra grab 1, intra grab 2 and inter grabs 3 to 12. 95% confidence intervals are shown as error bars.



A test for normality showed that the data were not normally distributed (P=<0.005). Consequently a non-parametric Kruskal-Wallis test was carried out to investigate if there were any significant differences between the medians. The test showed that there were no significant differences between inter-grab and intra-grab sample medians (H = 11.46; DF = 19; P = 0.907; for reference, H is the Kruskal Wallis statistic and when compared to a table of critical values if it is greater than the critical value, with p<0.05 then there is a significant difference). Multivariate tests, completed using PRIMER version 6.1.5 (Clarke and Gorley, 2006), also indicate that PSDs are statistically indistinguishable, as demonstrated by results from SIMPROF and ANOSIM tests on the similarity measure, using Manhatten distance, between samples.

Figure 11.3 shows a clustering dendrogram of PSDs from Intra grab 1, Intra grab 2 and Inter grabs (3-12). The SIMPROF routine tests for a significant difference in similarity between pairs of samples and joins those that are indistinguishable with dotted red lines. Samples joined by solid black lines are those that are statistically different. While there is a significant difference between PSDs measured, these are mainly between Intra-grab 2 and the rest of the PSDs measured. These Intra-grab 2 PSDs are mostly present in one cluster (4 out of 5 samples) and all these contain relatively high proportions of 2mm fraction (2mm – 4mm) – 25%-34%, except for 1 replicate. Therefore while they are locally slightly different they are mostly consistent in their sediment type.

Figure 11.3 Clustering dendrogram of Intra and Inter grabs
Plotting the group average similarity between pairs of samples measured from Intra grab 1, Intra grab 2 and Inter grabs (3-12). Similarity calculated using full PSD data.



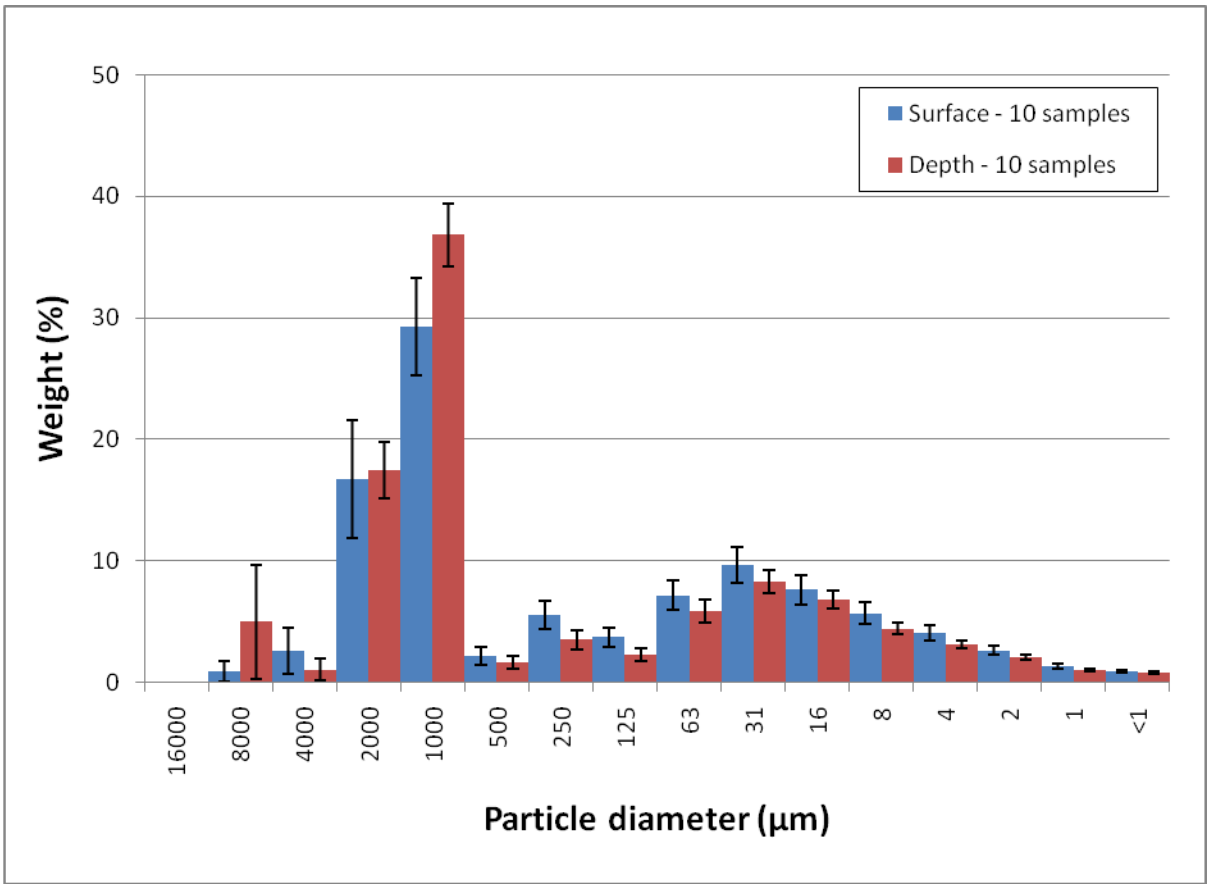
ANOSIM test results show that the Global R statistic values for tests comparing source of PSDs were small. On a scale of 0 to 1, a value of 0.247 is relatively small, indicative of a weak, almost negligible effect of source of sample (inter or intra) on the difference in similarity values between PSDs. Global R values closer to 1 would have indicated that the source of the PSD was significantly dissimilar.

b/ the effect of two methods of sample collection (2 cm surface scrape compared with 5cm depth integrated core) on the particle size distribution (PSD) measured

PSDs from surface sediments generally contain less gravel (6 of the 10 samples contain <20% gravel compared with 2 of the 10 samples for depth) and more silt/clay (mud) (%) (7 of the 10 samples contain >30% silt/clay compared with 2 of the 10 samples for depth).

The average surface PSD profile is compared to the average depth PSD profile in Figure 11.4. Generally the profiles are well matched, as is also indicated by the sediment descriptions already described.

Figure 11.4 Average particle size distributions (PSDs) of surface and depth samples. 95% confidence intervals are shown as error bars.

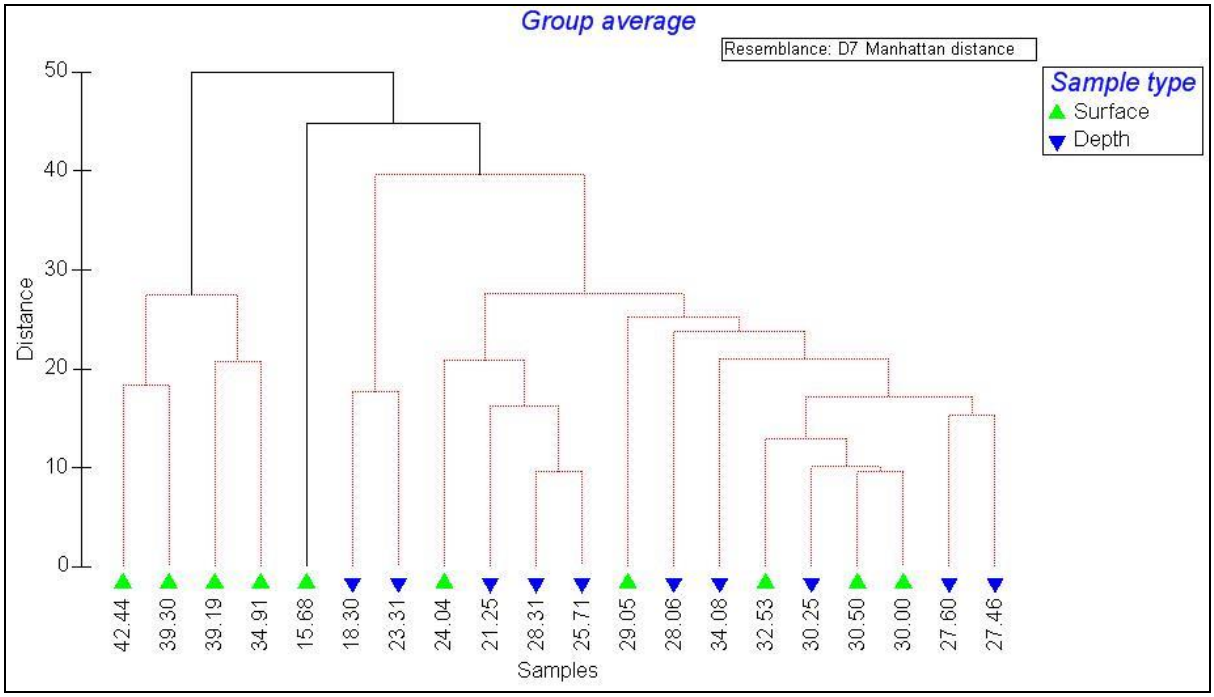


Surface PSD profiles are compared with depth PSD profiles using the following statistical analysis to determine significant differences. A test for normality showed that the data were not normally distributed (P=<0.005). Consequently a non-parametric Kruskal-Wallis test was carried out to investigate if there were any significant differences between the medians. The test showed that there were no significant differences observed (H = 5.78; DF = 10; P = 0.833 for reference).

Multivariate tests, completed using PRIMER version 6.1.5 (Clarke and Gorley, 2006), also indicate that PSDs are statistically indistinguishable. Figure 11.5 shows a clustering dendrogram of PSDs from surface and depth. The SIMPROF routine tests for a significant difference in similarity between pairs of samples and joins those that are indistinguishable

with dotted red lines. Samples joined by solid black lines are those that are statistically different. There is a cluster of four surface samples that are significantly different to the rest of the samples. These contain the highest proportion of silt/clay(%), as shown on the figure where silt/clay (%) values are shown as labels under the sample type symbol on the dendrogram.

Figure 11.5 Clustering dendrogram comparing surface and depth PSDs
Similarity calculated using full PSD data. Values of silt/clay(%) included under symbol for sample type.



ANOSIM test results show that the Global R statistic values for tests comparing source of PSDs were small. On a scale of 0 to 1, a value of 0.203 is relatively small, indicative of a weak, almost negligible effect of depth of sample (surface or depth) on the difference in similarity values between PSDs. Global R values closer to 1 would have indicated that the source of the PSD was significantly dissimilar.

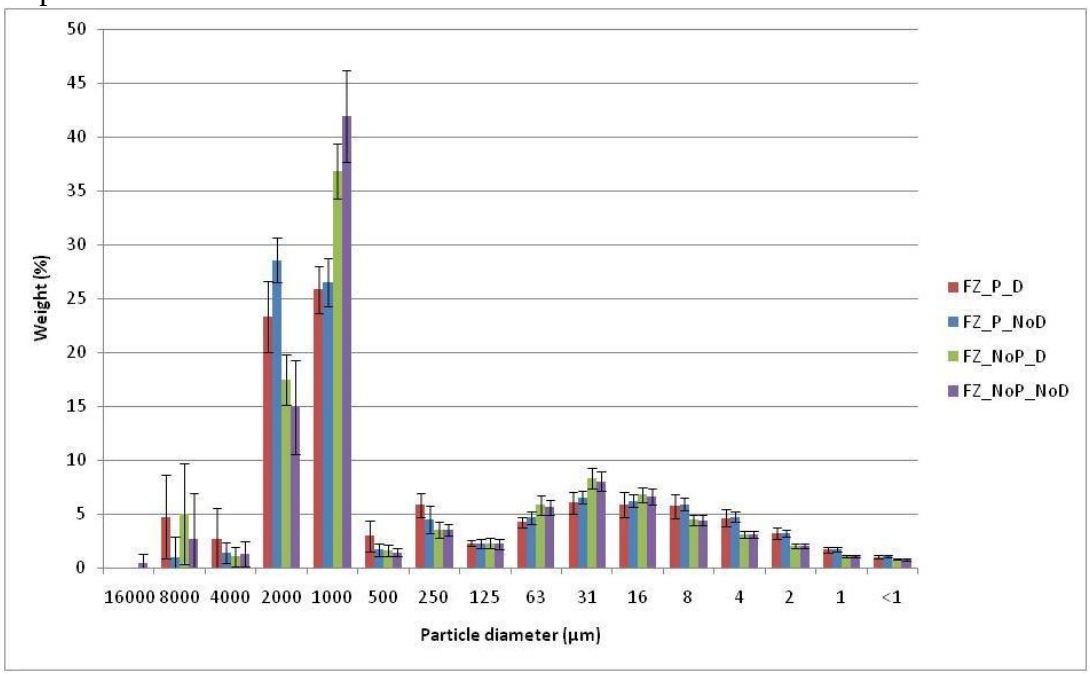
c/ the differences in the PSDs caused by freeze-drying after freezing compared with oven-drying after refrigeration, dispersant compared with no dispersant and organics removal compared with no organics removed.

The average PSD profiles for each experiment are shown in Figure 11.6. These show sediments pretreated by refrigeration then oven-drying are coarser than sediments that were frozen and freeze-dried.

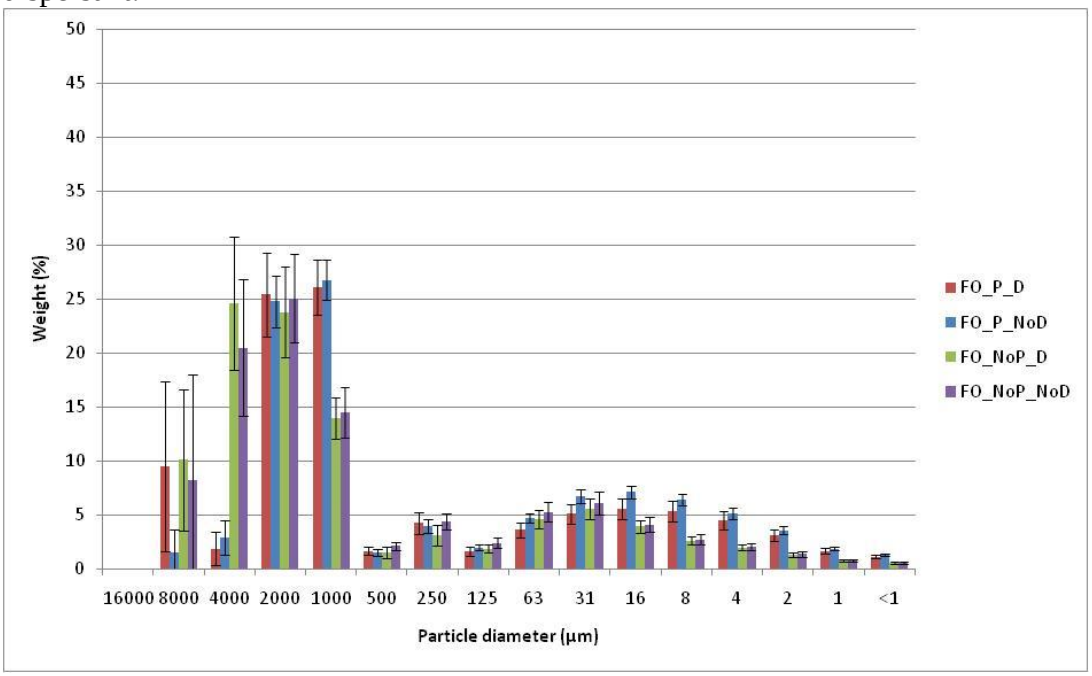
Figure 11.6 Average PSDs for NIEA experiment samples:
95% confidence intervals are shown as error bars.

Key –	FZ	Freeze and freeze-drying	FO	Fridge and oven
	P	Hydrogen peroxide added	NoP	No peroxide added
	D	Dispersant added	No D	Nodispersant added

a/ freezing and freeze-drying with hydrogen peroxide/no peroxide, and dispersant/ no dispersant.



b/ refrigeration and oven-drying with hydrogen peroxide/no peroxide, and dispersant/no dispersant.



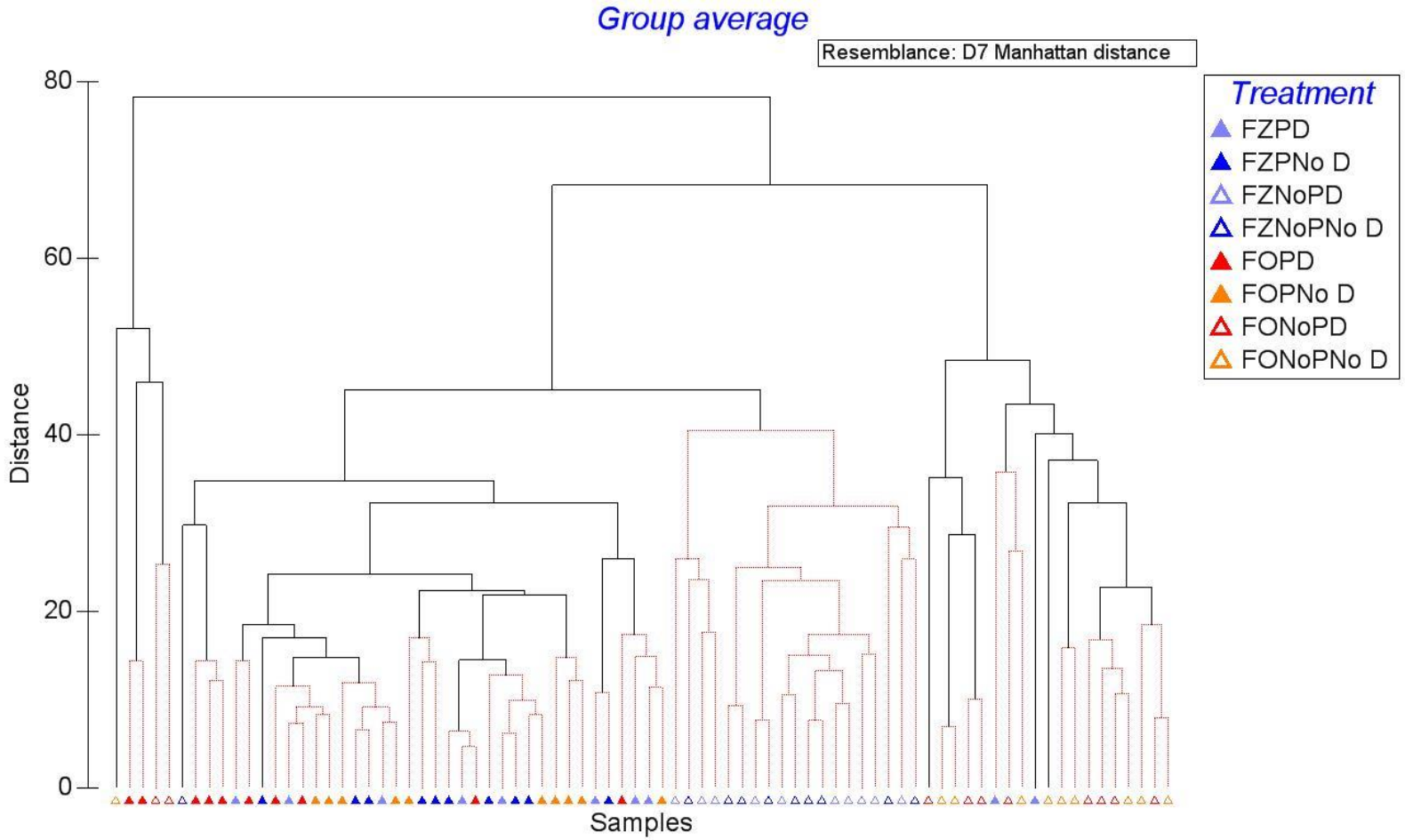
Experimental PSD profiles are tested using the following statistical analysis to determine significant differences. A test for normality showed that the data were not normally distributed (P=<0.005). Consequently a non-parametric Kruskal-Wallis test was carried out to investigate if there were any significant differences between the medians. None of the treatments were significantly different from one another (H = 5.75; DF = 7; P = 0.569 for reference).

Multivariate tests, completed using PRIMER version 6.1.5 (Clarke and Gorley, 2006), indicate there are some significant differences between PSDs. Figure B1.3 shows a clustering dendrogram of PSDs from all experiment samples, a/ labelled with freezing and freeze-drying or refrigeration and oven-drying, b/labelled with peroxide (hydrogen peroxide) or no peroxide, c/labelled with dispersant or no dispersant and d/labelled with all components of the treatment. SIMPROF routine tests for a significant difference in similarity between pairs of samples and joins those that are indistinguishable with dotted red lines. Samples joined by solid black lines are those that are statistically different.

The clustering dendrograms (Figure 11.7) show there is a clear separation between samples treated with hydrogen peroxide, and samples not treated with hydrogen peroxide; there is separation between samples that were frozen and freeze-dried, and samples that were refrigerated and oven-dried; and there is minimal difference between samples treated with dispersant, and samples not treated with dispersant as the samples are mixed within each cluster.

Figure 11.7 Clustering dendrogram for different pre-treatments

Plotting the group average similarity between pairs of samples measured from depth samples, labelled with freezing and freeze-drying (blue) or refrigeration and oven-drying (red/orange), with peroxide (hydrogen peroxide)% or no peroxide+ , with dispersant (light blue or red) or no dispersant (blue or orange). Similarity calculated using full PSD data.



ANOSIM test results show that the Global R statistic value for tests comparing sample treatments on PSDs was slightly significant. On a scale of 0 to 1, a value of 0.493 is relatively significant and indicative that there treatments have effects on the difference in similarity values between PSDs. Global R values closer to 1 would have indicated that the source of the PSD was significantly dissimilar.

ANOSIM individual pairwise tests show that the most significant difference (R value of 0.94) is caused by refrigerating and oven drying compared with freezing and freeze-drying. This effect is reduced if hydrogen peroxide is applied to the sample, and there is no significant difference between refrigerating and oven dried samples that have been pre-treated with hydrogen peroxide, compared with freezing and freeze-drying that have been pre-treated with hydrogen peroxide.

11.1.1.4 Recommendations

a/ Inter v intra PSD

The results show that at this CSEMP temporal monitoring site (with gravelly muddy sands and muddy sandy gravels) there are negligible differences between the source of sediment (from within the same grab or separate grabs) for measurement of PSD. The advantage of not taking sediment for PSA from the biological sample means there is no loss of biology (within the sediment sample removed). This supports the original recommendation in the Green Book for taking a sediment sample from a grab separate to the biology grab.

b/ surface v depth-integrated PSD

The results show that at this site, used for CSEMP temporal monitoring, there are negligible differences between surface and depth measurements of PSD.

The disadvantage of not measuring PSA from the surface (contaminant sample) as well as the depth (biological sample) means differences in silt/clay% would not be adequately represented. Most surface samples at this site contained more silt/clay % (32% +/- 8) compared with depth samples with less silt/clay% (26% +/- 4.5).

c/ the differences in the PSDs caused by freezedrying after freezing compared with oven-drying after refrigeration, dispersant compared with no dispersant and organics removal compared with no organics removed.

The results show that at this site, used for CSEMP temporal monitoring, there are slight differences caused by different pre-treatments to measurements of PSD. Oven-drying is known to aggregate particles, and the PSDs are coarser as a result, as shown in these experiments.

Treatment of samples with hydrogen peroxide to remove organics caused differences in PSDs measured compared with samples not pre-treated with hydrogen peroxide in this case.

Treatment of samples with dispersant did not cause differences in PSDs measured compared with samples not pre-treated.

11.1.2 Cefas

11.1.2.1 Introduction

The Centre for Environment and Fisheries and Aquaculture Science (Cefas) completed the following experiments to produce evidence in support of recommendations for PS methodology in support of biological analysis for the NMBAQC.

Aims tested:

a/ the effect of the source of the sediment sample (separate grab or same grab) on the particle size distribution (PSD) measured.

b/ the effect of two methods of sample collection (2 cm surface scrape compared with 5cm depth integrated core) on the PSD measured.

c/ differences in the particle size distributions (PSDs) caused by organics removal compared with no organics removed, as well as laser diffraction analysis of fine sediment (<63μm) compared with pipette analysis.

11.1.2.2 Methods

Tests a and b

Five 0.1m^2 day grabs with one depth integrated sample and one surface scrape were collected as well as five separate 0.1m^2 day grabs (primarily collected for biological samples) each with one depth integrated sample (representing PSD of biological samples) from eight CSEMP sites. Samples were collected on Cefas Endeavour in July 2009.

Depth integrated cores were collected with a cut-off syringe (3cm diameter) which was inserted vertically into grab sediment to the depth of the grab, at least 5cm giving approximately 15ml of sample removed. Surface scrapes were removed using a stainless steel spoon to a maximum depth of 2cm, achieving 100ml of sample.

All samples were frozen after collection at sea, and stored at -18 to -20˚C as soon as they were returned to the laboratory.

Each sample was analysed directly using laser diffraction, by a Malvern Mastersizer 2000, after defrosting. The sample was added to the Hydro G section of Malvern Mastersizer until obscuration reached between 15 to 20%. Measurement cycles commenced following 20 seconds of ultrasound.

Test c

Representative subsamples of wet fine sediment (<63μm) from 22 samples collected on Cefas Endeavour in June 2009 from the East coast (North Sea) were analysed by laser diffraction, and by pipette analysis, first with organic removal using hydrogen peroxide and secondly, without organic removal. Exact methodology available from Rob Nunny, Ambios Environmental Consultants Ltd.

11.1.2.3 Results

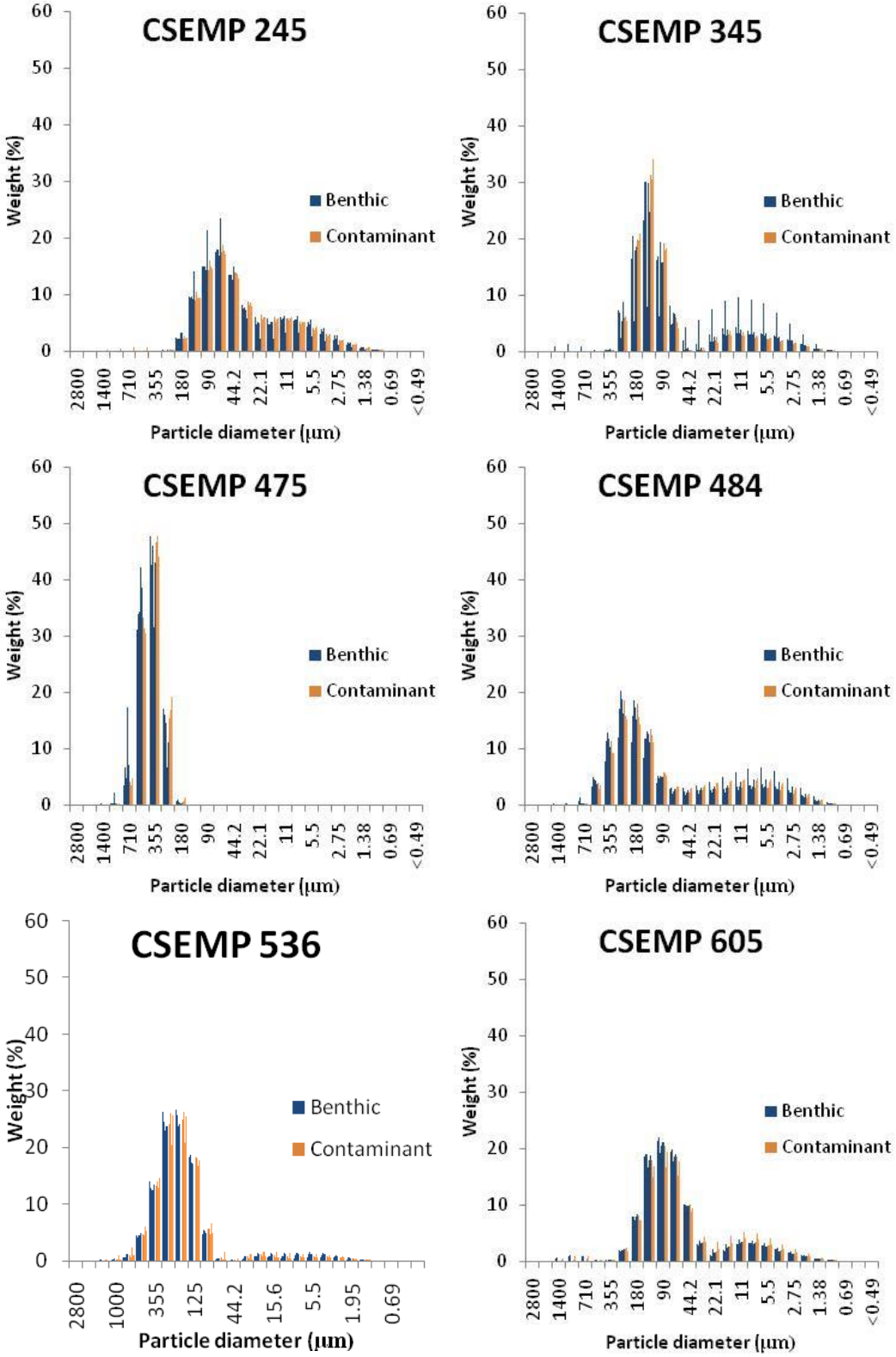
Tests a and b

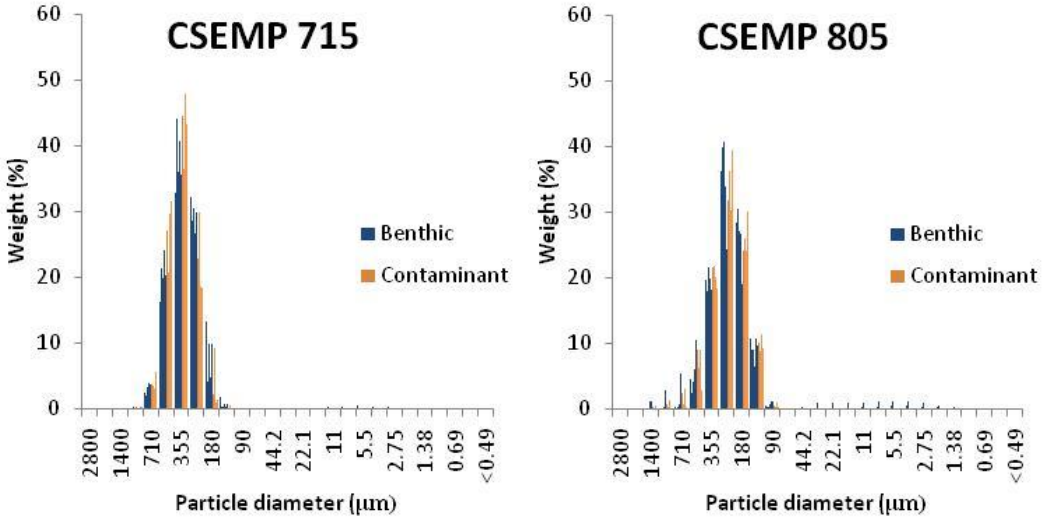
The sediments are a mixture of muddy sands, sandy muds and sands.

a/ the effect of the source of the sediment sample (separate grab or same grab) on the PSD measured.

PSDs of depth integrated cores from benthic and contaminant grabs at CSEMP sites are shown in Figure 11.8. These clearly show the similarity of PSD profiles for each site, regardless of the source of sample (benthic or contaminant grab).

Figure 11.8 Depth integrated PSDs from benthic and contaminant grabs CSEMP sites 2009



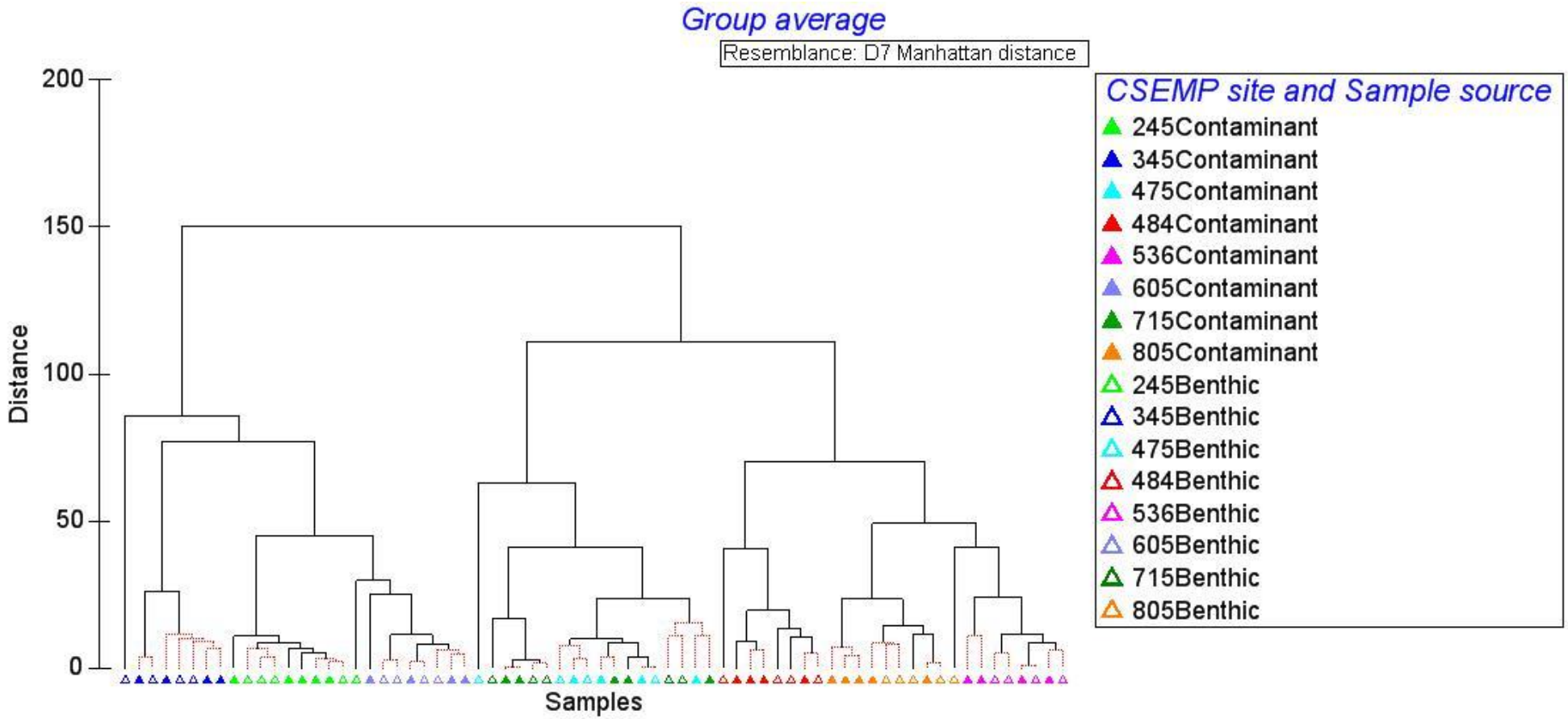


Benthic PSD profiles are compared with contaminant PSD profiles using the following statistical analysis to determine significant differences between each particle diameter measured within the PSD profile. A test for normality was completed and if this showed that the data were not normally distributed (P=<0.005), a non-parametric Kruskal-Wallis test was carried out to investigate if there were any significant differences between the medians. A two-sample T-test was completed if the data was normally distributed. Overall only 11 of a possible 216 fractions tested showed significant differences between benthic and contaminant samples (within site CSEMP 245 (15.6µm and 22.1µm,); CEFAS 345 (1.95µm, 2.75µm, 3.9µm, 5.5µm, and 7.8µm); CSEMP 475 (710µm and 1000µm); CSEMP 484 (90µm) and CSEMP 715 (7.8µm)). There is no significant difference between benthic and contaminant samples for most of the CSEMP sites considered here.

Multivariate tests, completed using PRIMER version 6.1.5 (Clarke and Gorley, 2006) also indicate that PSDs are statistically indistinguishable, as demonstrated by results from SIMPROF and ANOSIM tests on the similarity measure, using Manhatten distance, between samples. Figure 11.9 shows a clustering dendrogram of PSDs from benthic and contaminant samples, a/ labelled with CSEMP site and b/labelled with sample source. The SIMPROF routine tests for a significant difference in similarity between pairs of samples and joins those that are indistinguishable with dotted red lines. Samples joined by solid black lines are those that are statistically different. There are seven significantly different clusters, a cluster for each CSEMP site, except two sites, CSEMP 475 and CSEMP 715, both described as medium sands which have merged to form one cluster. When the same dendrogram is labelled with sampling source, benthic or contaminant, it is clear there is a mix of each sample source in each cluster, and it is the CSEMP site that is responsible for producing the different clusters present, not the sample source.

Figure 11.9 Clustering dendrogram from benthic and contaminant samples

Plotting the group average similarity between pairs of samples measured from benthic and contaminant samples, labelled with CSEMP site (colours) and with sample source (%contaminant and +benthic). Similarity calculated using full PSD data.

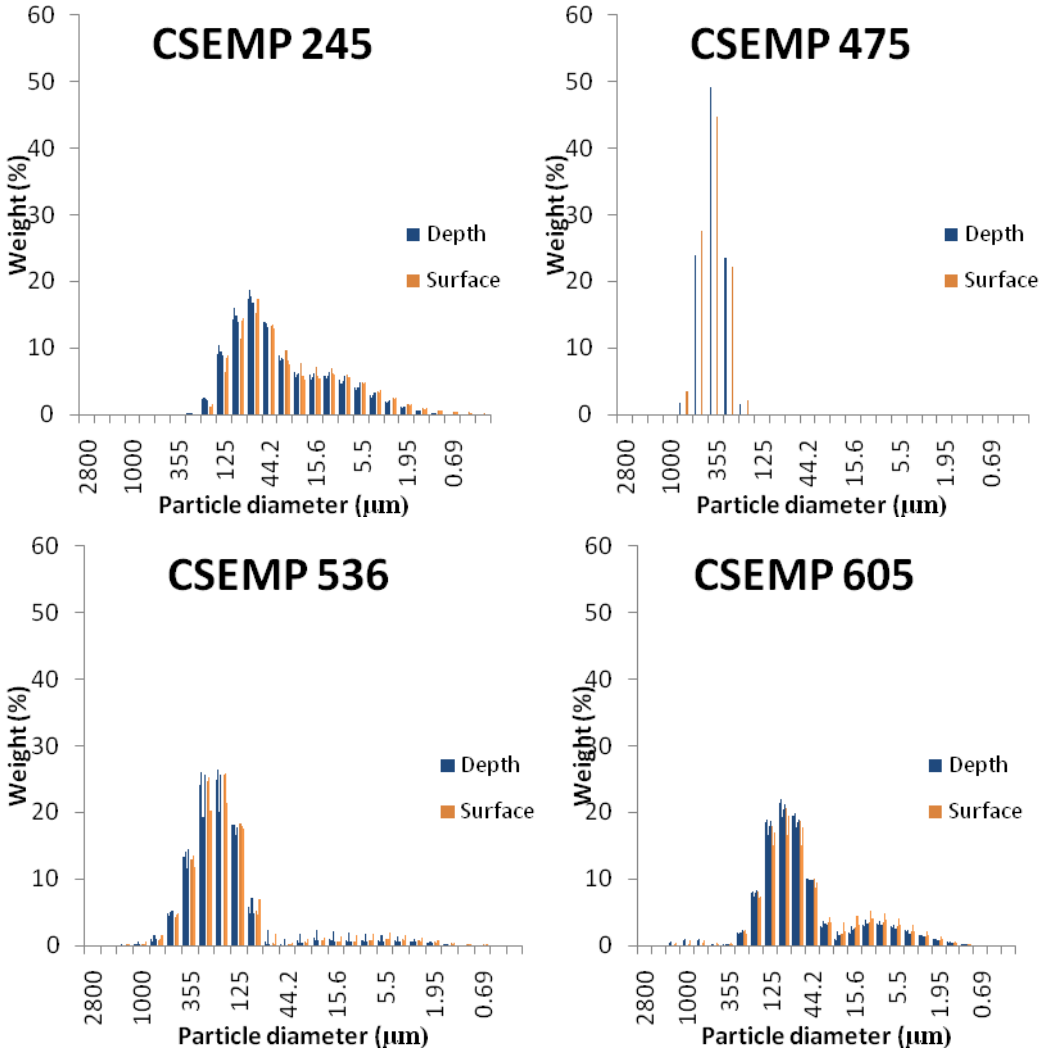


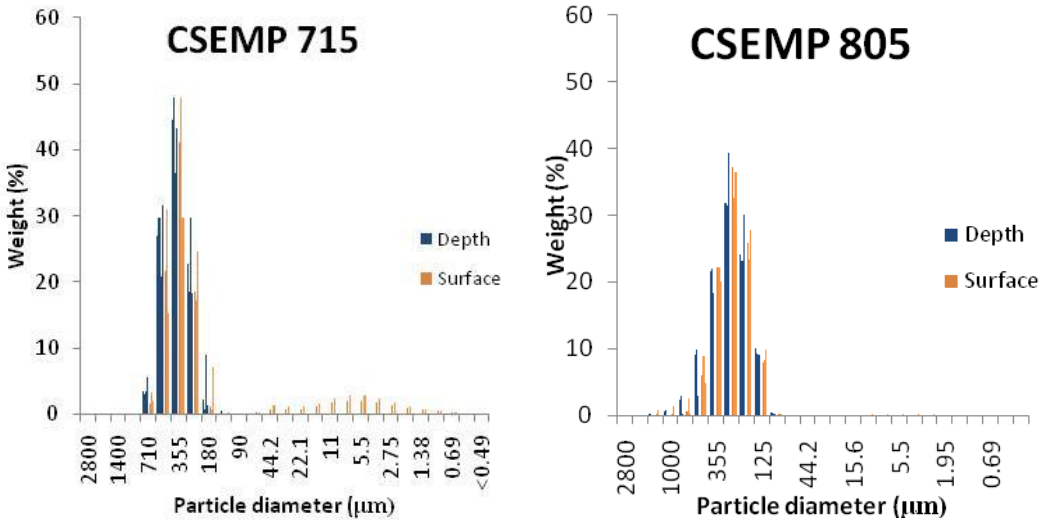
ANOSIM test results show that the Global R statistic values for tests comparing source of PSDs were small. On a scale of 0 to 1, a value of -0.024 is very small, indicative of a weak, almost negligible effect of source of sample (benthic or contaminant) on the difference in similarity values between PSDs. Global R values closer to 1 would have indicated that the source of the PSD was significantly dissimilar.

b/ the effect of two methods of sample collection (2 cm surface scrape compared with 5cm depth integrated core) on the PSD measured.

PSD profiles for surface and depth samples at these six CSEMP sites are shown in Figure 11.10. These clearly show the similarity of PSD profiles for each site, as well as indicating that for several sites the sediment contains more fine sediment for surface samples than for depth samples.

Figure 11.10 PSDs of surface and depth samples at six CSEMP sites 2009.
Each sample is represented for both depth and surface. Some sites had fewer measured results and so have fewer bars and these are paler.





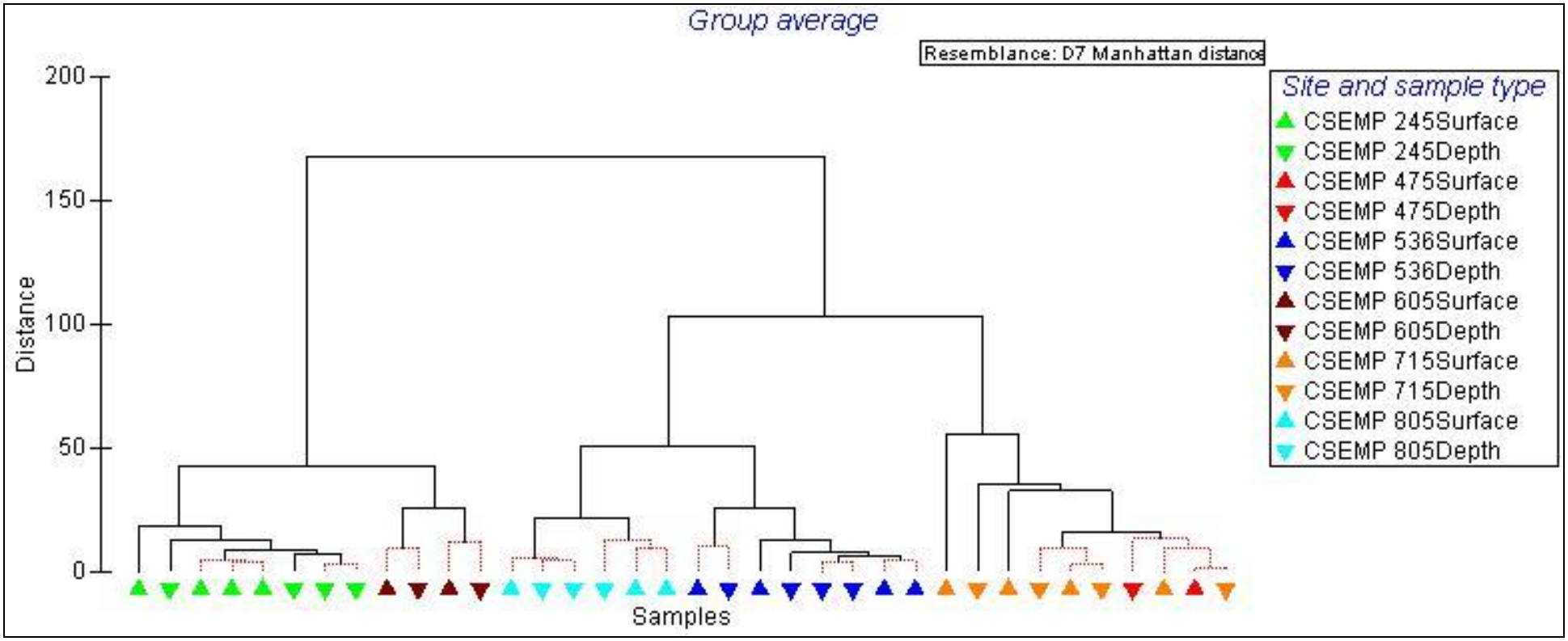
Surface PSD profiles are compared with depth PSD profiles using the following statistical analysis to determine significant differences between each particle diameter fraction measured within the PSD profile. A test for normality was completed and if this showed that the data were not normally distributed (P=<0.005), a non-parametric Kruskal-Wallis test was carried out to investigate if there were any significant differences between the medians . A two-sample T-test was completed if the data was normally distributed. Overall 8 fractions of a possible 262 fractions tested showed significant differences between surface and depth PSDs (within site CSEMP 245 (1.38µm, 1.95µm, 2.75µm, 3.9µm and 180µm,); and CSEMP 536 (0.69µm, 0.98µm and 1.38µm).

Multivariate tests, completed using PRIMER version 6.1.5 (Clarke and Gorley, 2006), also indicate that PSDs are statistically indistinguishable, as demonstrated by results from SIMPROF and ANOSIM tests on the similarity measure, using Manhatten distance, between samples. Figure 11.11 shows a clustering dendrogram of PSDs from surface and depth samples labelled with CSEMP site and sample type (surface or depth). The SIMPROF routine tests for a significant difference in similarity between pairs of samples and joins those that are indistinguishable with dotted red lines. Samples joined by solid black lines are those that are statistically different. There are six clusters, a cluster for each CSEMP site, except two sites, CSEMP 475 and CSEMP 715, both described as medium sands which have merged to form one cluster.

Figure 11.11 shows these clusters are further subdivided at a lower level, and the surface samples are split from the depth samples for some sites (for example CSEMP 245 and CSEMP 536). For other sites there is a mix of sample types within each sub-cluster showing there are minimal differences between the sample type (surface or depth). This reflects that surface samples have slightly higher finer sediment content than depth samples, but these differences are small scale compared with the sediment type measured as the samples (surface and depth) cluster together for the same site, rather than between surface and depth.

Figure 11.11 Clustering dendrogram for surface and depth samples.
Plotting the group average similarity between pairs of samples measured from surface and depth samples.

Samples labelled with CSEMP site (different colours for each CSEMP site) and with sample type (surface % or depth &). Similarity calculated using full PSD data.



ANOSIM test results show that the Global R statistic values for tests comparing source of PSDs were small. On a scale of 0 to 1, a value of -0.04 is very small, indicative of a weak, almost negligible effect of source of sample (surface or depth) on the difference in similarity values between PSDs. Global R values closer to 1 would have indicated that the source, surface or depth, of the PSD was significantly dissimilar.

c/ differences in the particle size distributions (PSDs) caused by organics removal compared with no organics removed, as well as laser diffraction analysis of fine sediment (<63μm) compared with pipette analysis.

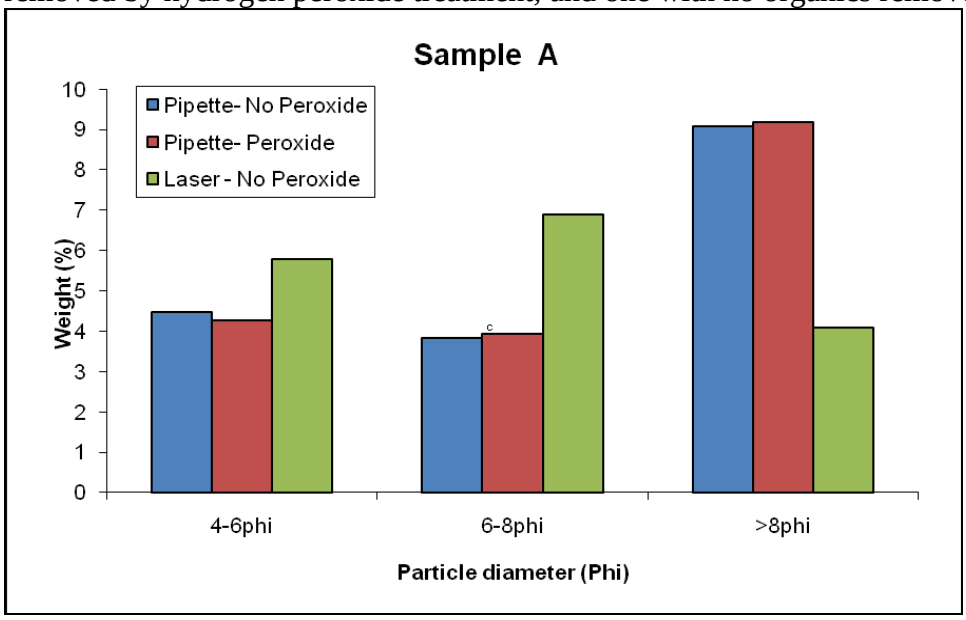
Results from two samples are presented to show the two patterns observed. The weights (%) for each of the following fractions, 4-6φ (very coarse silt), 6-8φ (fine and medium silt) and >8φ (very fine silt and clay), for each of the treatments and methods used are presented in Table 11.1. Each particle size distribution profile for these fractions is represented in Figure 11.12.

Table 11.1 Weights(%) of 4-6φ (very coarse silt), 6-8φ(fine and medium silt) and >8φ (very fine silt and clay) for Sample A and Sample B.

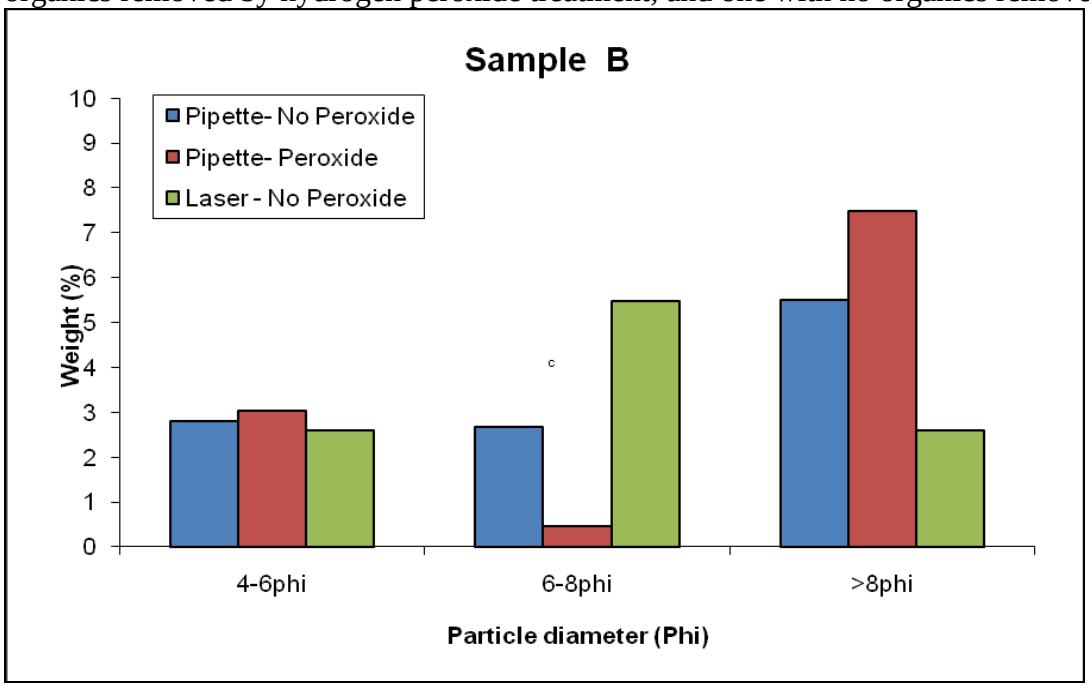
Sample	Method and pretreatment	Weight (%)		
		4-6 ϕ	6-8ϕ	>8ϕ
Sample A	Pipette- No Peroxide	4.47	3.84	9.08
Sample A	Pipette- Peroxide	4.27	3.94	9.18
Sample A	Laser - No Peroxide	5.78	6.89	4.09
Sample B	Pipette- No Peroxide	2.81	2.67	5.50
Sample B	Pipette- Peroxide	3.02	0.47	7.48
Sample B	Laser -No Peroxide	2.59	5.47	2.59

Figure 11.12 Comparison of pipette analysis and laser diffraction

a/ Bar chart of the fine fraction (>4φ (Phi) equivalent to <63µm) measured by pipette analysis and laser diffraction. Pipette analysis is completed on two subsamples, one with organics removed by hydrogen peroxide treatment, and one with no organics removed.



b/ Bar chart of the fine fraction (>4φ (Phi) equivalent to <63µm) measured by pipette analysis and laser diffraction. Pipette analysis is completed on two subsamples, one with organics removed by hydrogen peroxide treatment, and one with no organics removed.



Sample A shows there is minimal difference in results caused by removal of organics, compared with Sample B. Both Sample A and Sample B show there is an underestimation of the clay fraction by laser diffraction analysis compared with pipette analysis. Statistical significance is not tested as there are only two results presented.

11.1.2.4 Recommendations

a/ the effect of the source of the sediment sample (separate grab or same grab) on the PSD measured.

The results show that at these CSEMP sites, which are muddy sands, sandy muds and sands and are used for temporal monitoring as they are homogeneous and stable over time, there are negligible differences in the PSD between samples taken from the source of sediment for measurement of PSD.

b/ the effect of two methods of sample collection (2 cm surface scrape compared with 5cm depth integrated core) on the PSD measured.

The results show that at these CSEMP sites, muddy sands, sandy muds and sands, there are negligible differences between surface and depth measurements of PSD. However, silt/clay (%) for surface samples at most sites were higher than for depth samples.

c/ differences in the particle size distributions (PSDs) caused by organics removal compared with no organics removed, as well as laser diffraction analysis of fine sediment (<63μm) compared with pipette analysis.

Treatment of samples with hydrogen peroxide to remove organics can cause differences in PSDs measured compared with samples not pre-treated with hydrogen peroxide.

Laser diffraction methods underestimate clay content, as is shown when compared with results measured by pipette analysis.

11.1.3 NMBAQC PS Ring Test 23

11.1.3.1 Introduction

A summary report produced by David Hall is included giving the results of experiments testing organics removal compared with no organics removal, room temperature compared with refrigeration and freezing compared with refrigeration.

NMBAQC Scheme PS23 Extra Analyses Explanatory Notes

David Hall, Unicomarine Ltd.

December 2008

In the PS23 exercise (Scheme year 10) one laboratory (LB1002) returned data that showed a comparatively low percentage silt/clay fraction (Table 1). To ensure that the replicate sample received by LB1002 was not anomalous it was forwarded to the Scheme's laser technique analyst (University of Plymouth, Geography Department) for reanalysis. These results showed no deviation from the other replicates (Figure 3). Further subsamples, taken from the laser analyst's seven replicate samples, were analysed to investigate pre-treatment using hydrogen peroxide (Figure 4).

A short questionnaire was circulated to the PS23 participants to gather information regarding their sample storage methods (Table 2). As LB1002 appeared to be the only laboratory that had frozen PS23 upon receipt, the possibility of particle changes via freezing were measured (Figure 6) along with the effects of storage at room temperature for 6 weeks or refrigeration prior to analysis (Figure 5). The former tests were performed for both peroxide pre-treated and untreated subsamples.

Conclusions.

PS23 pre-treatment with hydrogen peroxide produced a ½ phi shift in the cumulative curve towards coarser material. Fine organic material has been removed from the analysis after pre-treatment.

PS23 pre-analysis storage at room temperature for six weeks shows no difference in results with samples refrigerated prior to analysis.

PS23 subsamples (both pre-treated with hydrogen peroxide and untreated) that were frozen prior to analysis showed no difference in results compared to subsamples refrigerated prior to analysis.

Note: These conclusions can only be stated for PS23 and cannot be applied to all marine mud sediments without extensive testing of additional sediments.

11.1.3.2 Recommendations

Treatment of samples with hydrogen peroxide to remove organics can cause differences in PSDs measured compared with samples not pre-treated with hydrogen peroxide. These results are supported by NIEA and Cefas experiments.

The results from this test suggest that freezing, refrigeration or keeping sediments at room temperature have minimal effect on the PSD measured.

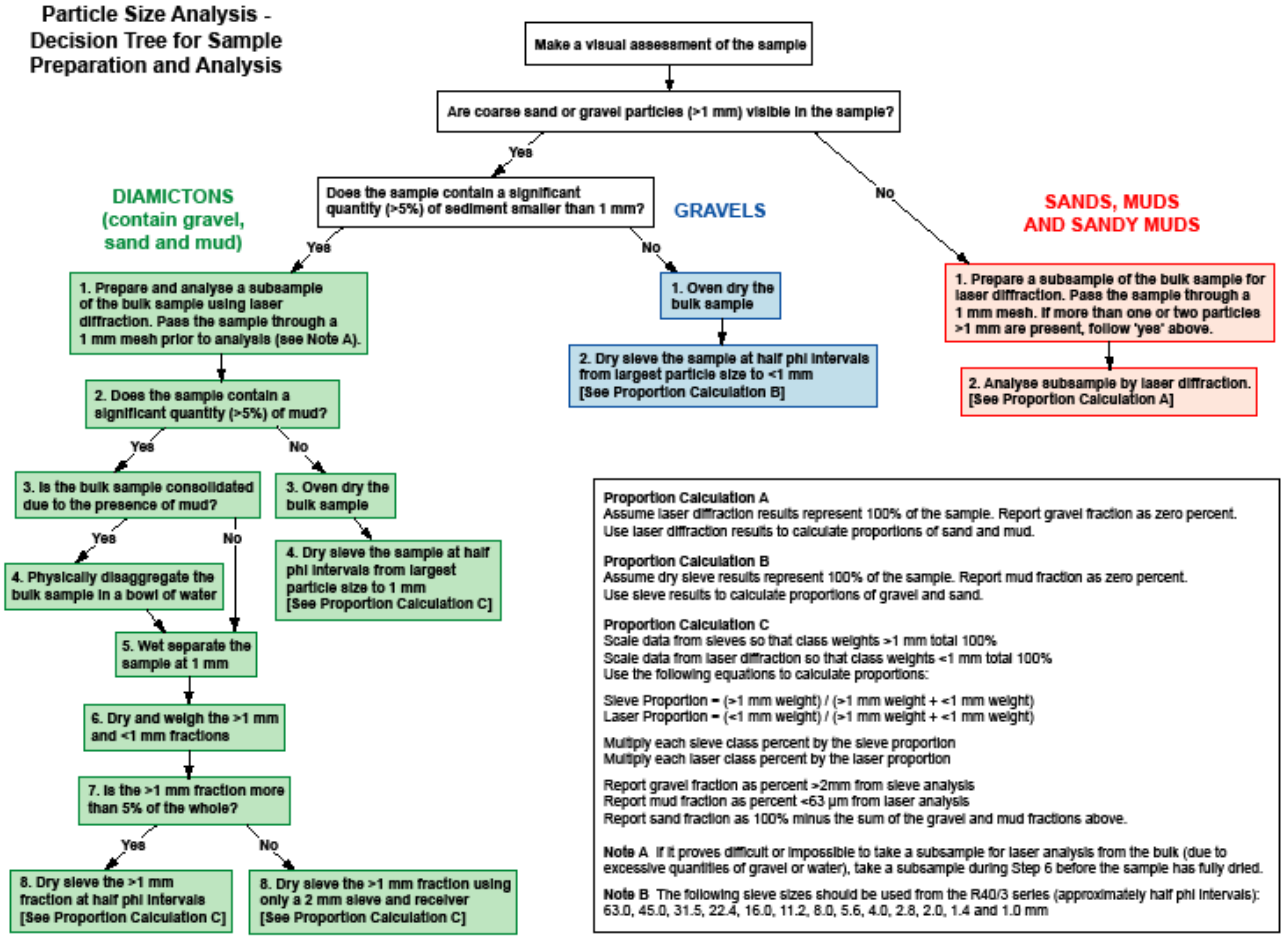
11.2 Background to standardised PS methodology

11.2.1 Flow chart defining PSA methods for sediment types (broken into three groups)

Ken Pye and Simon Blott (Ken Pye Associates Ltd) produced a flow chart (Figure 11.13) looking at the three main types of marine sediment that are encountered.

There are three sediment types identified as diamictons (mixed sediment), gravels, and sands (sands, muddy sands and sandy muds). If all the survey samples were gravels then sieving would be most appropriate, and if all the survey samples were sands then laser diffraction methods would be most appropriate. Diamictons require both sieve and laser methods. While CSEMP samples are predominantly sands, it is likely there will be a requirement to

Figure 11.13 PSA methodology based on three sediment types
(provided by Ken Pye and Simon Blott for NMBAQC sediment methodology workshop July 2009).



11.2.2 Photographs showing steps for completion of recommended PSA method

1/ Remove a subsample for laser analysis

CARE – this needs to be a representative subsample.
Photo A- Stirring sample to homogenise. If there is a lot of water on the top of the sample – remove before homogenising.
Photo B- Removing subsample and placing in a pot. It is important this sample is representative of the whole sample.

Photo A:

Photo B:

2/ Complete laser analysis on the <1mm fraction

A 1mm screen is advised before allowing sediment into the laser sizer.
Any laser data >1mm is discounted (see in stage 5), but if coarser material gets into the laser sizer it may cause damage.

Measure at least 3 replicates of the <1mm fraction using the laser sizer.

Photo C-1mm screen
Photo D- Emptying sample onto 1mm screen
Photo E- Washing sediment through the 1mm screen – USE as little water as possible
Photo F- Placing a subsample of <1mm into the laser sizer.

Photo C:

Photo D:

Photo E:

Photo F:

3 / Wet split the remaining sediment over a 1mm sieve

Take the rest of the sample and split at 1mm. This can be done using a wet sieve shaker or just placing a sieve over a bucket. The sample is placed on sieve and material <1mm is washed through.
Photo G: Wet sieve shaker
Photo H: 1mm sieve over a 5 litre plastic bucket.

Photo G:

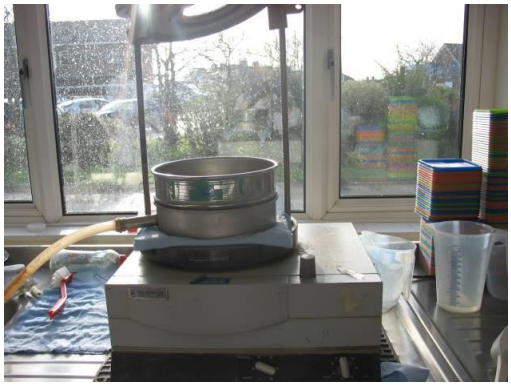
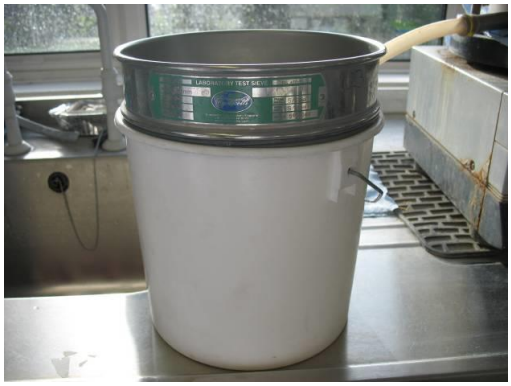


Photo H:



4/ Dry sieve >1mm fraction at 0.5φ intervals. Record the weight of any material <1mm

Sediment >1mm is oven dried and then dry sieved at 0.5φ intervals.
Sediment <1mm (after splitting in part 3) is left to settle out, and then any water siphoned away. This sediment is then dried in the oven and the weight <1mm is recorded.

Photo I: Dry sieve stack

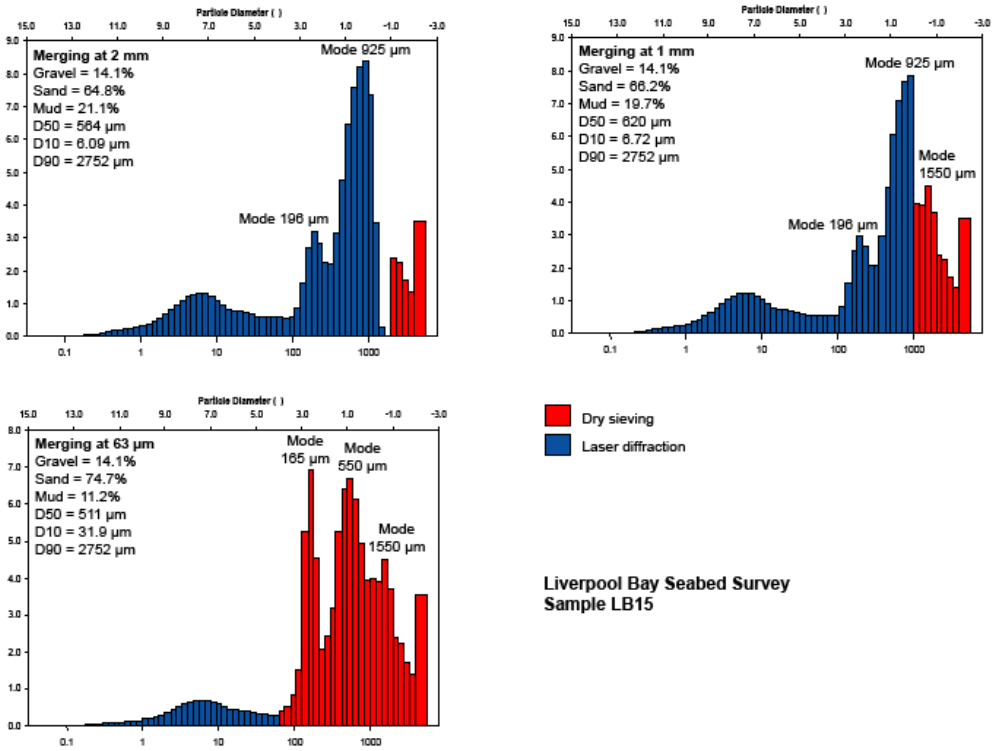
Photo I:

11.2.3 Sieve and laser comparisons to show merging issues between these two methods

The following 4 examples of sediments were provided by Ken Pye and Simon Blott (Ken Pye Associates Ltd.), using both sieve and laser methods, and then merged in 3 different ways (at 63µm, at 1mm and at 2mm).

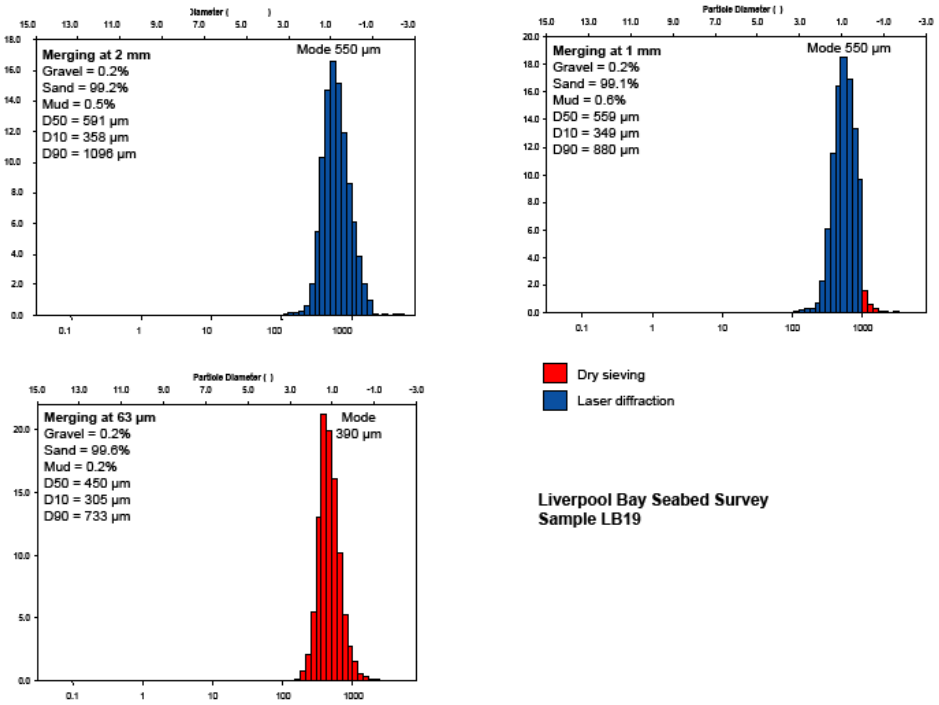
Example 1: Liverpool Bay Seabed Survey Sample LB15

Merging at 2mm results in a gap in the distribution and means the mode 1500µm is not recorded. The sieve data has modes of 165µm compared with 195µm for laser data, and at 550µm compared with 925µm showing that the laser sizer measures the same particles bigger than is recorded by sieves



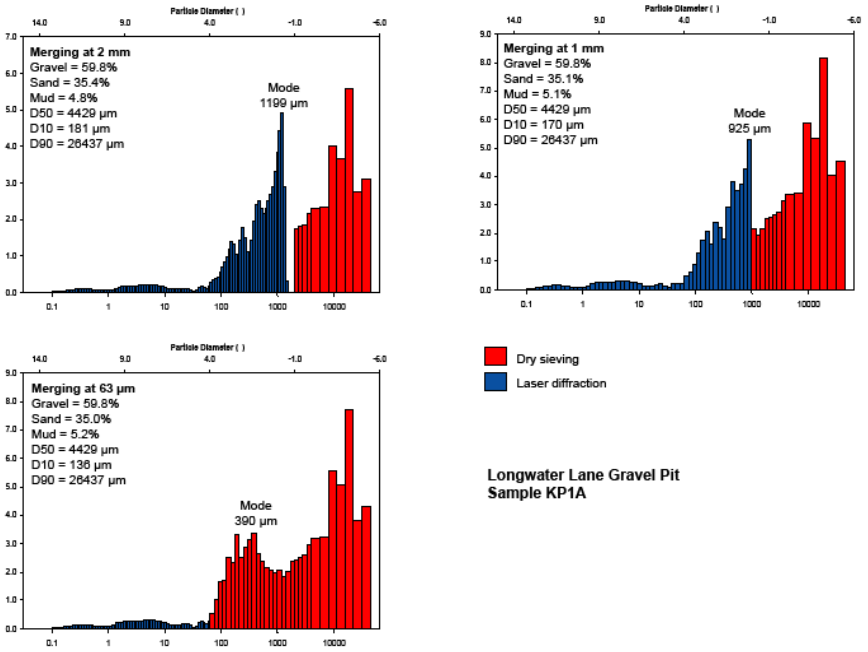
Example 2: Liverpool Bay Seabed Survey Sample LB19

The sieve data has a mode of 390µm compared with 550µm for laser data showing that the laser sizer measures the same particles bigger than is recorded by sieves



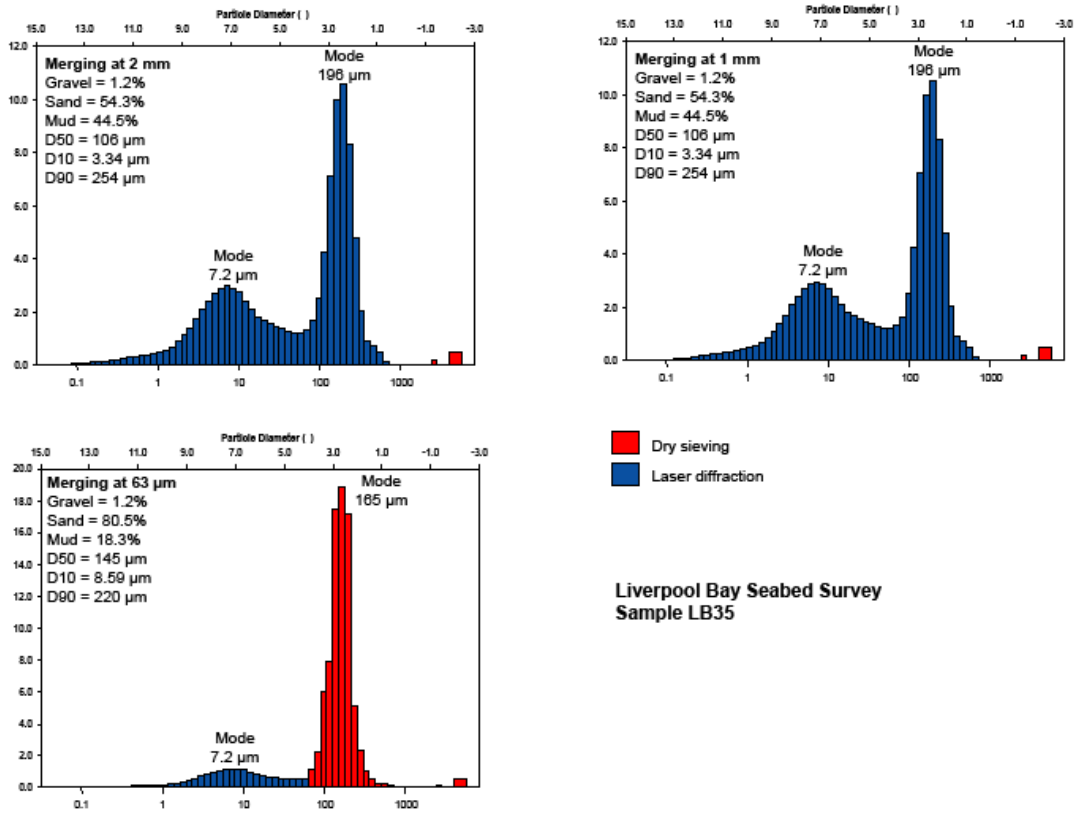
Example 3:Longwater Lane Gravel Pit Sample KP1A

Merging at 2mm results in a gap in the PSD. The sieve data has a mode of 390µm compared with 925µm for laser data screened at 1mm, and 1100µm for laser data screened at 2mm showing that the laser sizer measures the same particles bigger than is recorded by sieves.



Example 4: Liverpool Bay Seabed Survey Sample LB35

The sieve data has a mode of 165µm compared with 196µm for laser data showing that the laser sizer measures the same particles bigger than is recorded by sieves.



11.2.4 Worked examples for merging sieve and laser data for use in recommended PSA method

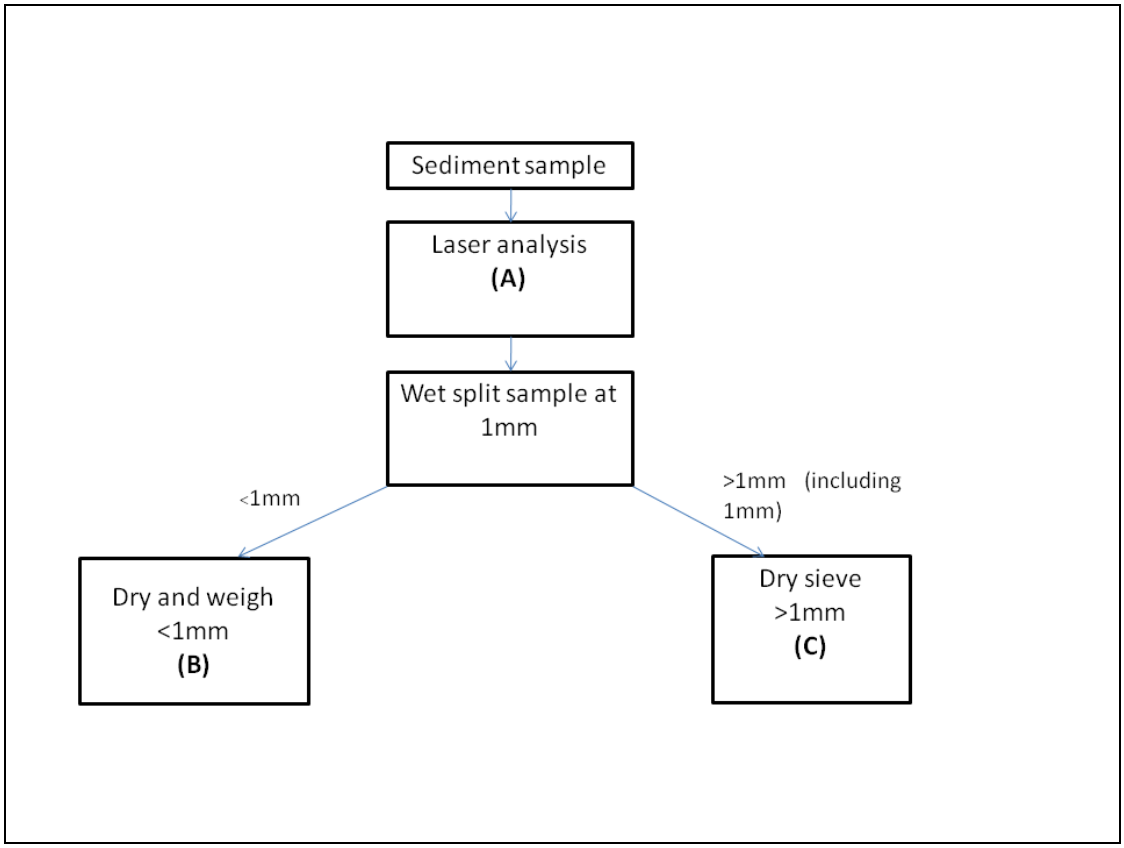
The following example is based on the spreadsheet sent out for the NMBAQC PSA method test (with some extra clarifications). The flow chart presented in
Figure 11.14 shows there are three data elements (A, B and C) required to merge the sieve and laser data together to produce a full PS distribution.

In this example, laser data is exported at 0.5 ϕ intervals, but this could also be done at 0.25ϕ intervals to give increased resolution.

The three data elements are:
Laser data (**A** on
Figure 11.14)
Total weight of <1mm sediment after wet sieving (**B** on
Figure 11.14)
Sieve data (**C** on
Figure 11.14)

Sieve data is measured as a weight (g) at 0.5ϕ intervals. The weight of sediment above the sieve is recorded. Please note that sediment collected in the pan during dry sieving should be added to <1mm sediment as is shown below.

Figure 11.14 Flow chart showing data elements to merge sieve and laser data



An example data set based on results submitted for the NMBAQC PSA method test is presented in Table 11.2- laser data (A) raw data, Table 11.3 -total weight of <1mm sediment after wet sieving (B)) and Table 11.4 -sieve data (C).

Table 11.2 Laser data (A) - Raw laser data.
For TEST3, some of the sample was measured >1mm (only 88.55% of the laser distribution is <1mm).

	Phi	Diameter (µm)	TEST 1	TEST2	TEST3
Volume (%)	0.5	710.00	4.93	18.05	14.31
	1.0	500.00	14.20	26.40	18.67
	1.5	355.00	20.65	25.66	17.67
	2.0	250.00	20.85	15.22	14.17
	2.5	180.00	12.68	4.18	9.12
	3.0	125.00	5.28	0.47	6.17
	3.5	90.00	0.94	0.59	2.86
	4.0	63.00	0.94	1.51	1.29
	4.5	45.00	1.48	1.14	0.46
	5.0	31.25	1.44	0.47	0.35
	5.5	22.10	1.05	0.22	0.37
	6.0	15.63	1.24	0.50	0.39
	6.5	11.05	1.82	0.88	0.40
	7.0	7.81	2.44	1.16	0.44
	7.5	5.52	2.58	1.16	0.49
	8.0	3.91	2.29	0.96	0.50
	8.5	2.76	1.72	0.66	0.44
	9.0	1.95	1.29	0.43	0.31
	9.5	1.38	0.76	0.22	0.13
	10.0	0.98	0.52	0.09	0.00
	10.5	0.69	0.46	0.03	0.00
	11.0	0.49	0.35	0.00	0.00
	>11	<0.49	0.10	0.00	0.00
	Check = 100	TOTAL	100.00	100.00	88.55

Table 11.3 Total weight of <1mm sediment after wet sieving (B)

Sample	**Barcode**	<1mm Foil tray (g)	<1mm Foil tray and dried sediment (g)	<1mm dried sediment (g)
TEST 1		46.00	536.00	490.00
TEST 2		46.00	513.00	467.00
TEST 3		46.00	531.00	485.00

Table 11.4 Sieve data (C)

	Phi	Diameter (µm)	TEST 1	TEST2	TEST3
Weight of sediment above the sieve (g)	-6	63000	0.00	0.00	0.00
	-5.5	45000	0.00	0.00	0.00
	-5	31500	0.00	0.00	0.00
	-4.5	22400	2.22	0.00	7.96
	-4	16000	9.13	0.00	51.81
	-3.5	11200	28.37	20.02	91.82
	-3	8000	32.82	47.78	86.18
	-2.5	5600	47.52	38.36	79.46
	-2	4000	43.66	22.38	57.86
	-1.5	2800	50.63	14.90	42.78
	-1	2000	53.64	15.12	37.90
	-0.5	1400	61.86	20.20	28.20
	0	1000	62.82	40.58	29.80
		<1000µm (PAN)	51.35	10.86	15.64

11.2.4.1 Normalise the laser data

The laser data is normalised so that everything <1mm adds to 100%. In Table 11.2, TEST 3 adds up to 88%, the rest of the sample was measured as being >1mm. In Table 11.5, laser data for TEST 3 has been normalised to 100%.

Table 11.5 Laser data normalised so that all <1mm laser data adds up to 100.

	Phi	Diameter (μm)	TEST 1	TEST2	TEST3
Volume (%)	0.5	710.00	4.93	18.05	16.16
	1.0	500.00	14.20	26.40	21.08
	1.5	355.00	20.65	25.66	19.96
	2.0	250.00	20.85	15.22	16.00
	2.5	180.00	12.68	4.18	10.30
	3.0	125.00	5.28	0.47	6.97
	3.5	90.00	0.94	0.59	3.23
	4.0	63.00	0.94	1.51	1.46
	4.5	45.00	1.48	1.14	0.52
	5.0	31.25	1.44	0.47	0.40
	5.5	22.10	1.05	0.22	0.42
	6.0	15.63	1.24	0.50	0.44
	6.5	11.05	1.82	0.88	0.46
	7.0	7.81	2.44	1.16	0.50
	7.5	5.52	2.58	1.16	0.56
	8.0	3.91	2.29	0.96	0.57
	8.5	2.76	1.72	0.66	0.50
	9.0	1.95	1.29	0.43	0.35
	9.5	1.38	0.76	0.22	0.14
	10.0	0.98	0.52	0.09	0.00
	10.5	0.69	0.46	0.03	0.00
	11.0	0.49	0.35	0.00	0.00
	>11	<0.49	0.10	0.00	0.00
	Check = 100	TOTAL	100.00	100.00	100.00

11.2.4.2 Calculate total <1mm (g)

Add sieve pan weight (from sieve data(C)) to total weight of <1mm sediment after wet sieving (B) as shown in Table 11.6.

Table 11.6 Total dried weight of <1mm(g) added to <1mm sediment in sieve pan (g)

Sample	Dried <1mm sediment(g)	<1mm from dry sieve pan (g)	**TOTAL <1mm(g)**
TEST 1	490.00	51.35	541.35
TEST 2	467.00	10.86	477.86
TEST 3	485.00	15.64	500.64

11.2.5 Convert laser volume (%) data into weights (g)

The laser volume (%) data is converted into weights (g) using the total weight <1mm (g) as shown in Table 11.7

Table 11.7 Laser data converted from volume (%) to weight (g) using total <1mm (g)
Sieve data is included in grey.

	Phi	Diameter (µm)	TEST 1	TEST2	TEST3
Weight (g)	-6	63000	0.00	0.00	0.00
	-5.5	45000	0.00	0.00	0.00
	-5	31500	0.00	0.00	0.00
	-4.5	22400	2.22	0.00	7.96
	-4	16000	9.13	0.00	51.81
	-3.5	11200	28.37	20.02	91.82
	-3	8000	32.82	47.78	86.18
	-2.5	5600	47.52	38.36	79.46
	-2	4000	43.66	22.38	57.86
	-1.5	2800	50.63	14.90	42.78
	-1	2000	53.64	15.12	37.90
	-0.5	1400	61.86	20.20	28.20
	0	1000	62.82	40.58	29.80
	0.5	710	26.71	86.27	80.89
	1.0	500	76.85	126.14	105.54
	1.5	355	111.79	122.63	99.92
	2.0	250	112.89	72.71	80.09
	2.5	180	68.66	19.97	51.56
	3.0	125	28.58	2.25	34.90
	3.5	90	5.07	2.82	16.16
	4.0	63	5.09	7.22	7.29
	4.5	45	8.01	5.45	2.62
	5.0	31.25	7.81	2.26	1.99
	5.5	22.10	5.70	1.04	2.10
	6.0	15.63	6.73	2.37	2.21
	6.5	11.05	9.83	4.21	2.28
	7.0	7.81	13.19	5.53	2.50
	7.5	5.52	13.95	5.54	2.78
	8.0	3.91	12.38	4.60	2.84
	8.5	2.76	9.33	3.15	2.49
	9.0	1.95	6.97	2.05	1.78
	9.5	1.38	4.11	1.04	0.71
	10.0	0.98	2.82	0.45	0.00
	10.5	0.69	2.47	0.16	0.00
	11.0	0.49	1.88	0.00	0.00
	>11	<0.49	0.52	0.00	0.00
		TOTAL (>1mm Sieve)	392.67	219.34	513.77
		TOTAL (<1mm)	541.35	477.86	500.64
		TOTAL	934.02	697.20	1014.41

11.2.5.1 Calculate percentage merged PS distribution

The weights (g) for both sieve and laser data are divided by the total weight for each fraction to produce a merged PS distribution (Table 11.8).

Table 11.8 Merged PS distribution

	Phi	Diameter (µm)	TEST 1	TEST2	TEST3
Percentage (%)	-6	63000	0.00	0.00	0.00
	-5.5	45000	0.00	0.00	0.00
	-5	31500	0.00	0.00	0.00
	-4.5	22400	0.24	0.00	0.78
	-4	16000	0.98	0.00	5.11
	-3.5	11200	3.04	2.87	9.05
	-3	8000	3.51	6.85	8.50
	-2.5	5600	5.09	5.50	7.83
	-2	4000	4.67	3.21	5.70
	-1.5	2800	5.42	2.14	4.22
	-1	2000	5.74	2.17	3.74
	-0.5	1400	6.62	2.90	2.78
	0	1000	6.73	5.82	2.94
	0.5	710	2.86	12.37	7.97
	1.0	500	8.23	18.09	10.40
	1.5	355	11.97	17.59	9.85
	2.0	250	12.09	10.43	7.90
	2.5	180	7.35	2.86	5.08
	3.0	125	3.06	0.32	3.44
	3.5	90	0.54	0.40	1.59
	4.0	63	0.54	1.03	0.72
	4.5	45	0.86	0.78	0.26
	5.0	31.25	0.84	0.32	0.20
	5.5	22.10	0.61	0.15	0.21
	6.0	15.63	0.72	0.34	0.22
	6.5	11.05	1.05	0.60	0.22
	7.0	7.81	1.41	0.79	0.25
	7.5	5.52	1.49	0.80	0.27
	8.0	3.91	1.33	0.66	0.28
	8.5	2.76	1.00	0.45	0.25
	9.0	1.95	0.75	0.29	0.18
	9.5	1.38	0.44	0.15	0.07
	10.0	0.98	0.30	0.06	0.00
	10.5	0.69	0.26	0.02	0.00
	11.0	0.49	0.20	0.00	0.00
	>11	<0.49	0.06	0.00	0.00
		TOTAL	100.00	100.00	100.00

The data and calculations for merging sieve and laser data are included in this Excel workbook: “Merging example dataset”.

Merging example dataset.xls

11.3 Worked examples of internal QC procedures for PSA

11.3.1 QC of sieve data

The weight of the sediment prior to sieving can be checked with the weight measured during the sieving process, which in turn can be checked with the weight of the sediment after sieving. There should be less than 5% difference between them overall, although for small samples the error is higher.

Table 11.9 Sieving checks

Sample	Sediment (in sieves) (g) = SIE	Presieving (g) = PRE	Post-sieving (g) = POST	PRE - SIE (g)	PRE -POST (g)	POST - SIE (g)	% Difference (POST-SIE/SIE) X100)
1	35.83	36.02	35.95	0.19	0.07	0.12	0.33
2	92.15	92.28	92.23	0.13	0.05	0.08	0.09
3	70.44	70.51	70.54	0.07	-0.03	0.10	0.14
4	85.74	85.83	85.74	0.09	0.09	0.00	0.00
5	72.75	72.84	72.83	0.09	0.01	0.08	0.11

11.3.2 Use of Internal reference standards for QC

A sand standard has been developed in Cefas as a quality assurance reference for completing daily checks for laser sizer measurements. Figure 11.15 shows the standard sand particle size distribution profiles for sand measurements completed from 3/3/2010 to 9/5/2010. Table 11.10 shows the coefficient of variation values for the standard sand. A coefficient of variation (CV) of d (0.1), d(0.5) and d(0.9) of less than 3% is defined in ISO 133020 as an indication of good repeatability. Please note that in reality 3% is on the low side and greater variability being expected for natural sediment samples – a maximum of 20% (based on 3 replicates being measured) should be used as a guide. Figure 11.16 shows a control chart for the standard sand, using the d(0.5) as the measure of variation between measurements, and using 2 X standard deviation to set an upper and lower limit.

Figure 11.15 Standard sand reference PSDs

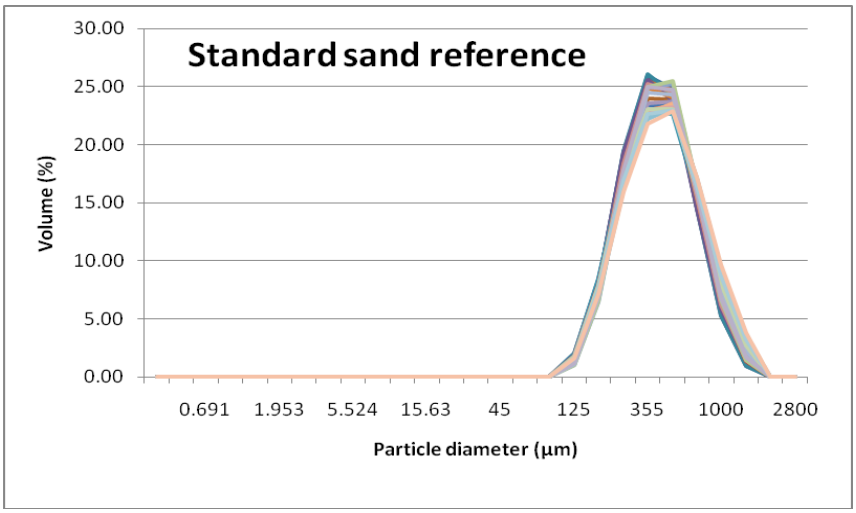
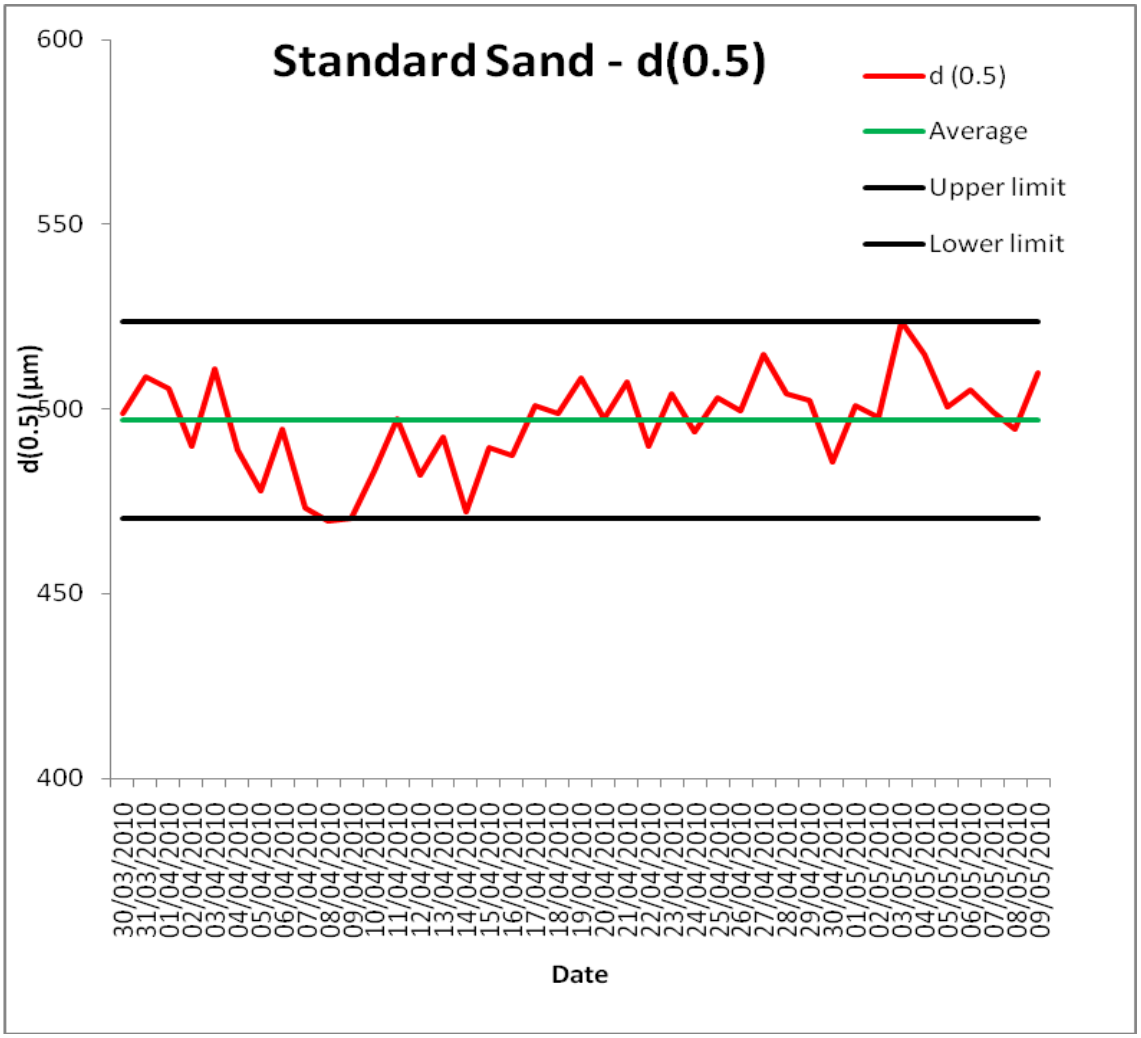


Table 11.10 Coefficient of variation (%) values for standard sand reference

Standard sand	Mode	d (0.1)	d (0.5)	d (0.9)
Average	487.99	256.05	496.33	987.59
Standard deviation	13.76	4.77	12.60	50.14
Upper limit (average +2Xstdev)	515.52	265.59	521.54	1087.87
Lower limit (average =2Xstdev)	460.47	246.50	471.12	887.31
Coefficient of variation	2.82	1.86	2.54	5.08

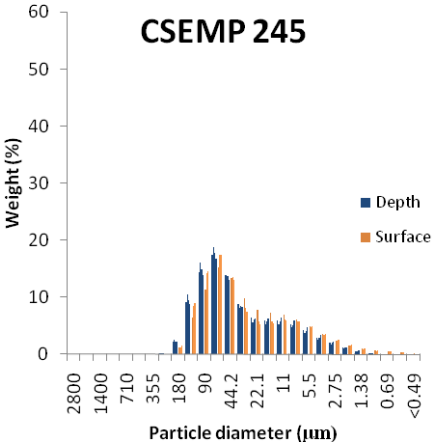
Figure 11.16 Standard sand reference control chart for d(0.5)
Limits are defined in Table 11.10

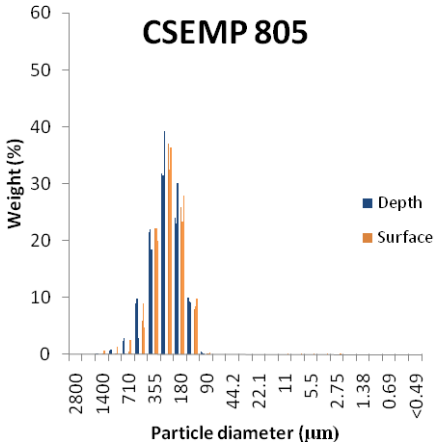


11.3.3 Verification of PS results using photographs and visual description completed at time of sample collection

PS results can be cross-referenced to sample photographs and visual descriptions completed when samples are collected. This should be a first measure of verification, and can only be an approximate check. Two examples of sediment sample photographs taken at point of collection are given in Figure 11.17. The sediment PSD profiles and descriptions match well with the photographs given.

Figure 11.17 Verification of PS results using sediment photographs
Examples collected for CSEMP on Cefas Endeavour CEND10/09: CSEMP245 and CSEMP 805 with measured particle size distribution histograms





All sediments (four replicates) at CSEMP 245 are described as very fine sandy, very coarse silt. Sediments at CSEMP 805 are described as unimodal medium sand.

11.4 NMBAQC PSA Laboratory workshop notes - 7th December 2017

Copy of workshop notes circulated following the NMBAQC PSA Laboratory workshop held on 7th December 2017 at National Laboratory Services (NLS) laboratory in Leeds. Recommendations have been added in the main text where appropriate.

Participation in NMBAQC PSA scheme since the introduction of standardised PSA methodology demonstrates marked improvement in comparability of PS data. Introduction of PS own sample test has assisted further in supporting PS method standardisation.

The 2017 PSA workshop aimed to refine NMBAQC PSA methodology further, progressing issues that had arisen, primarily with laser analysis.

Summary notes and actions

1. Subsampling – dispersion techniques

The sample needs to be fully dispersed, prior and during laser analysis.
There are minimal adverse effects if a chemical dispersant is used, and their use can help with clay/sticky samples.
Therefore, based on the evidence in this workshop, the guidance will be updated to indicate use of a chemical dispersant is acceptable. Details of dispersant concentration and amounts must be included within sample metadata to allow measurement comparability.
Use of ultrasonics for dispersion is already included in the Guidance but will be updated to indicate ultrasonic use during laser analysis (as well as before) will help minimise agglomeration of particles.

2. Laser analysis differences

Replicates

The Guidance indicates usefulness of completing three analysis runs per test sample to check stability of sample during laser analysis, as well as three replicate sub-samples of each sample to check subsample representativeness, resulting in nine results for one sample. This is to help support setting up of methodology and has been updated to indicate that once the method is established (sample is stable and subsample representativeness is reproducible) then less replicates are required. It is expected that a minimum of 1 duplicate subsample for 1 in 10 samples must be completed.

Subsample representativeness, as evidenced at the workshop, is the most likely problem that will be encountered, particularly with muddy sands. Use of higher obscurations (20% is highest recommended obscuration for polydisperse samples when using Malvern Mastersizer 3000, and 15% for Beckman-Coulter 13320 laser sizer) is proposed to help ensure sand is measured in muddy sand samples but also noting that the higher the obscuration, the more chance there is of multi-scattering effects (which mean diffraction pattern will become confused and artefacts will be introduced).

Minimum laser run must be at least 60 seconds as broad particle distributions present.

QC evidence

QC evidence should be provided alongside results data to demonstrate number of replicates completed, the variability of the replicates completed, how the final result was determined, use of certified reference materials and inhouse reference materials.

3. Instrument manufacturer differences between Beckman Coulter LS13320 and Malvern Mastersizer 3000

Three samples were circulated to Meritics Ltd (Beckman Coulter LS13-320), Malvern Instruments (Mastersizer 3000), and KPAL. Cefas and NLS also completed NMBAQC SOP analysis of these samples (3 subsamples only).

Sample	Brief sample description
S1	Mud (not pre-screened)
S2	Sand (pre-screened at 1mm)
S3	Slightly sandy mud (not pre-screened)

Separate tests for completion:
1. Manufacturer SOP
2. SOP as defined NMBAQC PSA guidance, including 3 replicates and 3 runs for each replicate (a suggested when setting up laser SOPs, but not explicitly expected for every sample.
3. As above but using dispersant – Addition of 1ml of 3% sodium hexametaphosphate (Calgon) to subsample. 3 replicates and 3 runs for each sample.

The main differences observed relate to the recorded percentages of silt and clay. While the overall mud (%) is similar between both instruments, the Mastersizer 3000 reports lower clay, and higher silt when compared to the Beckman-Coulter LS13-320 operated with the subsidiary PIDS system turned on.

Optical model

Optical models tested:
1/ Refractive index: Imaginary (absorption) index 0.1
2/ Refractive index: Imaginary (absorption) index 0.01
3/ Fraunhofer

Imaginary (absorption) index 0.01 produced strange artefacts (both instruments) and therefore should not be used. While 0.1 performs better, this model can also produce artefact peaks in the sub-micron range.

Fraunhofer proved to be the most comparable between the two laser sizers. Proposed to test this further using NMBAQC ring test samples (NLS, Cefas, and KPAL) to confirm this will reduce differences observed.

Another practical difference is the capability to measure bigger particles with the Beckman-Coulter LS13-320 when compared with the Mastersizer 3000, meaning screening of the laser sample is possible at 2mm rather than 1mm. Screening laser samples at 2mm (already included in the Guidance) rather than 1mm prior to laser analysis reduces artefacts produced when merging sieve and laser data.

In addition, the following topics were covered:

Sieve PS methodology compared to laser PS methodology

As demonstrated in previous workshops, sieve PS methodology (sieving to 63µm) gives a different PS distribution to that produced by laser dominated PS methodology. When NMBAQC participants first discussed PS methodologies, it was agreed a laser dominated PS methodology was required, and this is how we have arrived at the NMBAQC PS methodology as described. Use of shape factors to convert laser to sieve/and vice versa were presented. Many of the classifications used (eg Folk, etc) are based on sieving methods, and therefore laser based methods may well result in a different classification than they would have been if sieving methods had been used. While it is important to be aware of this, the PS data is being used to measure trends so if a consistent PS method is applied to all samples then this is achievable. Rather than modelling sieve data from laser data, it would be preferable to look at whether sieve based classifications can be updated with new thresholds to reflect new laser methodology. There is no doubt that sieving, particularly for coarser sand and gravel samples, is a good PS method and is still widely used. However, to sieve every sample (as well as wet screen sample) to 63µm would mean a lot of additional effort, potentially introduce confusion, and further complications. Sieving at 63µm to cross check laser method for muddy sands has merit. A comparative graph will be added to the Guidance to demonstrate the relationship between the >63µm and <63µm proportion when sieving compared with laser analysis.

Presence of Asbestos in sediment samples
Evidence of low levels of asbestos across different sources of sediments (ports, disposal site and offshore) and associated control procedures put in place to mitigate against this risk were highlighted. Further details are available. Please contact Claire Mason (claire.mason@cefas.co.uk).

11.5 NMBAQC PSA Data Standards workshop notes – 22nd June 2018

Copy of workshop notes circulated following the NMBAQC PSA Data Standards workshop held on 22nd June 2018 at Eco Innovation Centre, Peterborough. Recommendations have been added in the main text where appropriate.

Summary notes

A/ Data

- Different size fractions reported for laser analysis - driven by databases
- Differences also driven by client requirements
- Signposting to most detailed (Raw) data (Unique code)
- Metadata not routinely reported - Collate existing fields &templates
- Use of Gradistat (Blott and Pye,2001) to calculate statistics (Blott, S.J. and Pye, K. (2001) GRADISTAT: a grain size distribution and statistics package for the analysis of unconsolidated sediments. Earth Surface Processes and Landforms 26, 1237-1248)
- Data template could include a standards metadata field
- How do different choices (metadata) affect data comparability

- Cost associated with reporting metadata has to be proportionate
- MEDIN guidelines for metadata (Grab and core) looking to add PSA fields
- Can record raw data file for laser? (Metadata within file) - readable? Can database store additional files - cheaper
- Lab and field processes are separate - understanding is needed
- When subsampling in field -if water is present can be difficult to subsample fines
- Image analysis of sediments - auto match to data - does it look sensible? – linked to QC
- Sabellaria reef - how best to analyse sample
- Sieve shaker can pulverise sediments (for example, sabellaria tubes)
- PS is measured to support biological and/or contaminant interpretation- so may have different metadata fields
- Standard reporting of laser data
- Mud proportion affected by approach - sieve vs laser – important to understand particularly in relation to temporal studies
- Methodology may differ depending on use of data, but want to be balanced to make sure data can be used for wide range of purposes potentially
- Depth of sample should be recorded in metadata
- Presence of Lithology/shell/organics should be recorded
- Age & how sample stored should be recorded
- Different approaches for summary statistics etc.
- Graphical output - size spectrum
- Report confidence (repeat analysis, internal/external)
- Number of decimal places queried - 2 decimal places is recommended for PS data

B/ Metadata

- Look to already available metadata guidelines (DarwinCore, Medin, EMODnet)
- NERC has reference lists for equipment that would be of value to retain details such as Laser sampler, sieve size etc
- Producers may only get a subset of data so cannot retain all information
- I.e. there are 3 levels of producer identified (surveyor, laboratory and person who brings it all together)
- Often the lab only gets a subset of the information because it is “sensitive”, so they cannot be responsible for compiling all metadata
- This requires a “chain of custody” guidance, so it is clear who is responsible for what, and prevent certain metadata being lost.
- There was also some ambiguity regarding who is the point of storage (laboratory/ client/ archive), and for how long and guidance on this would be part of a chain of custody.

- it would be good practice for the laboratory to be given basic information on location to support their data insurance practices, as they may know if there are odd results for an area where it would not be expected.
- Photos are worth archiving. But there are many types of photograph (in situ, on deck, on sieve, etc.) so there is an agreement that this is worth retaining somewhere but not sure how. Possibly in link in data to the survey report or spreadsheet. Worth further thought. There is a cost and time to retain and generate this metadata, and how much is it used once sample is accepted? Some organisational SOP.
- Thought that some clients only ask for specifics from lab. But if there was guidance then sometimes they may ask for more and become more consistent because they would know what the industry standard is.
- No samples need to be retained and this could be part of chain of custody doc. Also not only where there are no samples but the attributed reason for the no sample (weather conditions, substrate, cobble in grab, malfunction etc). Metadata needed but not retained.
- Depth of sample/depth of subsample. As some surface scrapes are taken.
- SOP of named procedure for taking subsample. This would be in a report but is often lost from the data by the time it is archived and this would affect the comparability of results.
- Cobbles are a problem. And are dealt with in different ways. Visual assessment of the coarse fraction is sometimes used to ensure the complete sample is taken. As a subset of cobbles may not be feasible to sample without skewing results.
- Similarly need volume of whole grab in metadata.
- Cobble analysis is common. And this can be factored into samples. However, this is regularly lost and would be solved by chain of custody.
- Record when the sample was analysed as well as when was it collected?

C/ Quality (QC –quality control – completed in-house as part of routine method checks; QA – quality assurance – external quality assurance of sample analysis)

- Evidence of external QA through NMBAQC should include results not just participation
- Initial field sampling – needs to follow standard method
- Equipment and Methods - QC needs clarifying – laboratories vary in what QC is completed
- UKAS - need external accreditation with a pass/fail. Accreditation is lost if remedial action is not met. e.g. NMBAQC ring test.
- (JNCC) 5% of samples from each contract will go to external lab for QA across the range of sediment types measured.
- Reporting External QA data batch by batch is very useful for end users.
- ISO Standards exist for parts of the methods used (and are referred to – but often have been devised for different purposes (soils, pharmaceuticals, etc.)
- Re-certifying sieves optically (depending on your weave) - this is cheaper than a new sieve.

- Replicates - Error report, standard deviation – often details of replicates not included in final results
- Averaging repeats/first repeat. Averaging of results should only be used if replicates completed for every sample. 1st replicate should be used if, for example, 1 in 10 samples have replicates.
- Beckman-Coulter are producing new larger certified laser standards up to 1mm